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# ANNUAL REPORTS

Biological  
& Medical  
Serials

OF THE

# CHEMICAL LABORATORY

OF THE

# AMERICAN MEDICAL ASSOCIATION

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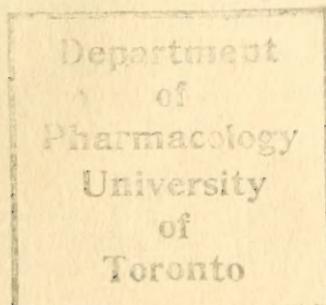
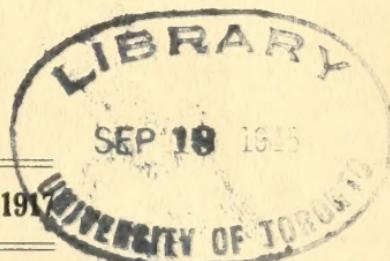
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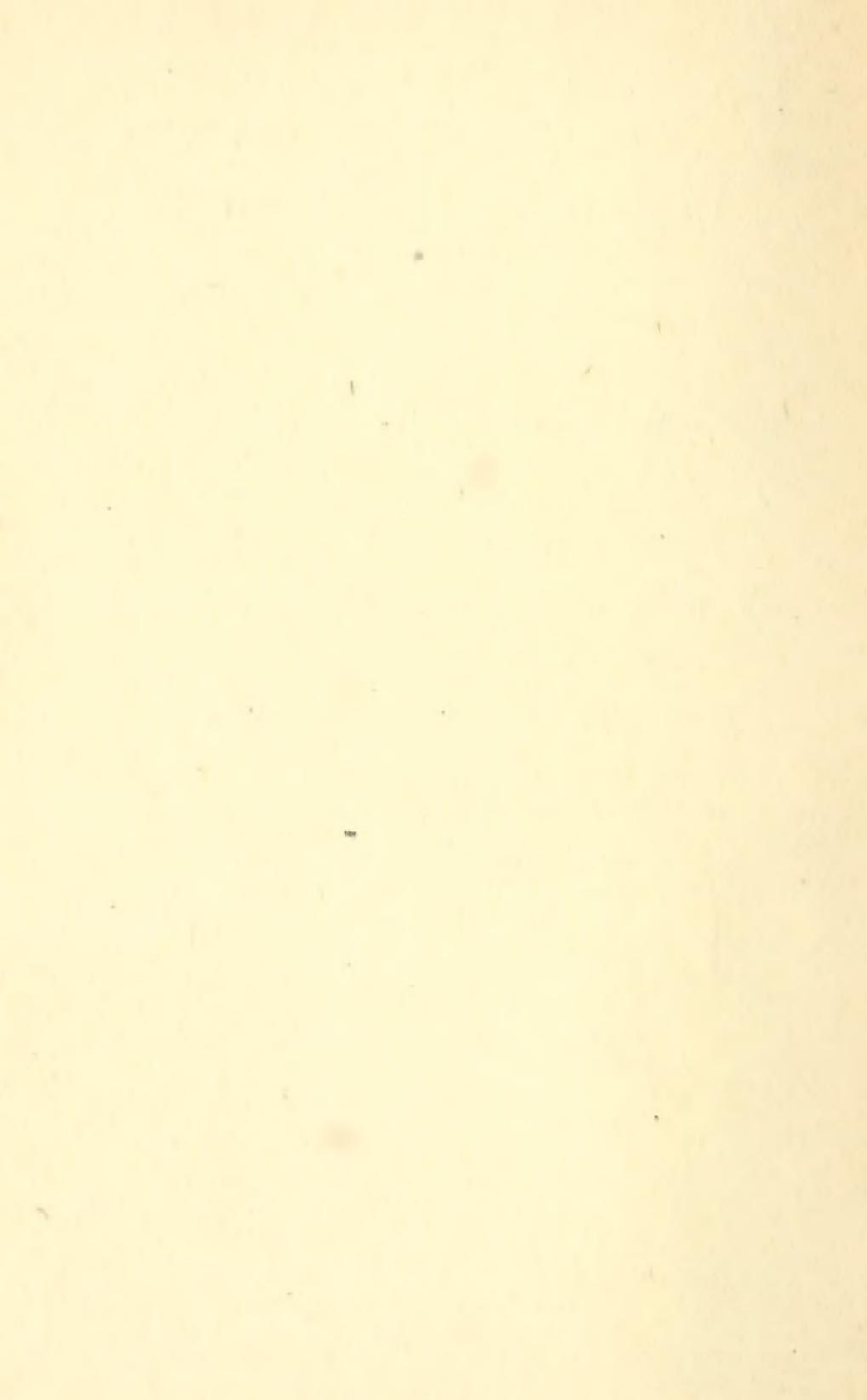
GENERAL INDEX TO VOLUMES 1-10 INCLUSIVE

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VOLUME 10. JAN.-DEC. 1915

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ANNUAL REPORTS  
OF THE  
CHEMICAL LABORATORY  
OF THE  
AMERICAN MEDICAL ASSOCIATION

VOLUME 10

JANUARY-DECEMBER, 1917

AND

GENERAL INDEX TO VOLUMES 1 TO 10, INCLUSIVE

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|---------------------|-------------------------------------|
| PART I. - - - - -   | REPRINT OF CONTRIBUTIONS            |
| PART II. - - -      | REPORTS ABSTRACTED FROM THE JOURNAL |
| PART III. - - - - - | REPORTS NOT PREVIOUSLY PUBLISHED    |

PRESS OF  
AMERICAN MEDICAL ASSOCIATION  
FIVE HUNDRED AND THIRTY-FIVE NORTH DEARBORN STREET  
CHICAGO



## PREFACE

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The present volume includes reports of the work of the past year, which has been carried out on the same lines as in previous years. The scope of this work is described in the preface of Volume II.

In addition to the work growing out of the investigations by the Council, the Laboratory's work includes the examination of "patent medicines" and a large amount of investigation of chemical questions connected with the Propaganda and the Queries and Minor Notes departments of THE JOURNAL of the American Medical Association.

In conformity with previous reports, the volume contains an account of those portions of the Laboratory's activities which it was thought would be of interest to drug analysts, i. e., those engaged in the examination of medicines.

In response to requests received from those who find the Annual Reports of this Laboratory of value for reference, a general index has been prepared for the ten volumes issued since the Laboratory's establishment in 1906, and is included with this volume.



Department  
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## PART I

### REPRINTS OF CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF THE AMERICAN MED- ICAL ASSOCIATION

#### NOTE ON THE DETERMINATION OF BORIC ACID BY TITRATION IN THE PRESENCE OF GLYCEROL

B. H. St. John, B.Sc.

(Reprinted from the *American Journal of Pharmacy*,  
January, 1917, p. 8)

The method usually given for the titration of boric acid is as follows:

Add methyl orange and neutralize with normal sulphuric acid, adding a few drops in excess, then boil to expel carbon dioxid, cool and exactly neutralize with tenth-normal sodium hydroxid volumetric solution. Enough glycerin is then added so that the solution shall contain at least 30 per cent. and the solution titrated to a pink color with phenolphthalein.

In the course of some determinations the author noted that in the neutralization to methyl orange with tenth-normal sodium hydroxid solution after boiling off the carbon dioxid, a very slow and unsatisfactory color change occurs. With the hope of obtaining a sharp color change methyl red was tried in place of the methyl orange. The change of color, as is usual with methyl red, was quite sharp and entirely satisfactory.

The necessity then arose of checking the accuracy of the results obtained by using methyl red in this titration. Accordingly a sample of boric acid was prepared by recrystallizing a commercial product from hot water. The crystals after being sucked as dry as possible on the filter were dried over concentrated sulphuric acid.

Four solutions were prepared by dissolving 0.2000 gm. of this pure boric acid in about 30 c.c. of water. To two of

these methyl orange was added, and to the other two methyl red. These solutions appeared acid to methyl red but alkaline to methyl orange. However, one drop (about 0.03 c.c. of tenth-normal alkali) sufficed to cause the methyl red to indicate a neutral state. And 0.10 c.c. of tenth-normal acid changed the methyl orange from the distinctly alkaline yellow color to the neutral tint. The sharpness of this color change with methyl orange seems to indicate that it is not the boric acid which causes the slow color change noted.

After the neutralization of the solutions, glycerin was added and the solution titrated to a pink color with phenolphthalein. The boric acid calculated from these titrations averaged 0.2007 gm. in the solutions neutralized to methyl orange; in those neutralized to methyl red, 0.2001 gm.

When glycerin is added to a solution containing methyl red the indicator changes its color to red and as the titration proceeds the red color fades slowly, the point at which the indicator is half transformed occurring when about half the alkali necessary for titration with phenolphthalein has been added.

TITRATION OF BORIC ACID IN SOLUTIONS  
CONTAINING SODIUM CARBONATE

Several solutions were prepared containing 1 gm. of sodium carbonate in each with a known amount of boric acid, thus simulating the conditions met with in the determination of boric acid. The titrations were carried out according to the method given below. The endpoints obtained with methyl red in the neutralization of excess acid were as sharp as usual but those obtained with methyl orange were, as the author had noted before, indistinct. Can it be that the endpoint of methyl orange is affected by a considerable amount of salts in the solution titrated? The results of these titrations are given in the attached table.

METHOD

The solution was carefully neutralized in the cold with normal sulphuric acid and about 0.2 to 0.3 c.c. of the acid added in excess, the beaker being covered with a watch glass meanwhile to avoid loss by effervescence. The solution in the beaker, still covered with the watch glass, was quickly

heated to boiling and boiled about 1 minute. In the case of the solutions in which methyl red was used as indicator, if the color had faded and become alkaline, normal acid was added until the red color was restored and the boiling continued for one minute. The solutions were then cooled to 20 to 25 C. and neutralized with tenth-normal sodium hydroxid volumetric solution. The neutral point with methyl orange being taken as that point where a clear yellow appears and all suggestion of pink or orange shade has disappeared. An equal volume of neutral glycerin was then added and the titration completed to the appearance of a pink color with phenolphthalein.

Boric acid taken gm.	Sodium carbonate taken gm.	$H_3BO_3$ found methyl orange gm.	Error per cent.	$H_3BO_3$ found methyl red gm.	Error per cent.
0.2000	1.0	0.2003	+0.15	0.1988	-.60
0.2000	1.0	0.2038	+1.9	0.2004	+.20
0.2000	1.0	0.2024	+1.2	0.2001	+.05
0.2000	1.0	0.2034	+1.7	.....	..
0.2500	1.0	0.2523	+.92	0.2497	-.12
0.2500	1.0	.....	....	0.2501	+.04
Average error.....			1.17		0.10

It is essential that care should be taken to cool the solution to room temperature before neutralizing as the indicators change color at a decidedly different hydrogen ion concentration when warm.

It will be seen that the results obtained by the use of methyl orange are high, while those obtained in the titrations in which methyl red was used, with one exception, are very close to the theoretical.

#### CONCLUSIONS

The results seem to indicate that methyl red can be used in place of methyl orange in the titration of boric acid, that it is more satisfactory since a sharp endpoint can be obtained, and that more accurate results can be obtained.

SOME COLOR REACTIONS OBTAINED FROM THE  
EXTRACT OF ACER SPICATUM (FALSE  
VIBURNUM OPULUS, VIBURNUM  
OPULUS U. S. P. VIII)

B. H. St. John, B.Sc.

(Reprinted from the American Journal of Pharmacy,  
January, 1917, p. 10)

Some two years since, while in the service of the Bureau of Chemistry, the author was engaged in the examination of a number of patent remedies which were claimed to be of value in the treatment of female troubles. Several of these were found to give Börntrager's test for emodin. However, other tests failed to classify the emodin-like material as any of the common emodin-bearing drugs, *i. e.*, aloes, cascara, rhubarb or senna.

Knowing the common use of the Viburnums in preparations designated for the treatment of female diseases, the idea suggested itself that an uncommon emodin-like substance might occur in the commercial drug or fluidextract, or perhaps was a common adulterant. Accordingly Börntrager's test was applied to such samples of commercial fluidextracts of *Viburnum opulus* and *Viburnum prunifolium* as were available.

The test was carried out in the following manner:

The fluidextract in a separator was diluted with about three volumes of water, about 5 c.c. of concentrated hydrochloric acid added, and the mixture shaken with about one fourth of its bulk of petroleum ether. After settling, the petroleum ether was decanted into a test tube containing 2 to 3 c.c. of 10 per cent. ammonia water, and the mixture allowed to stand, whereupon the crimson color appears at the juncture of the two liquids and gradually diffuses down into the ammonia. The fluidextract of *Viburnum prunifolium* gave only a faint yellow color, while the fluidextract of *Viburnum opulus* gave a very characteristic test.

Considering the possible adulteration of this fluidextract of *Viburnum opulus*, a sample of cramp bark-labeled *Viburnum Opulus U. S. P. VIII*—was obtained and a fluidextract prepared from it according to the U. S. P. VIII. (A portion of this sample was submitted to the Bureau's pharmacognocist for examination, but a report identifying this as *Acer spicatum* was not received until the author, then working in

the laboratory of the American Medical Association, had satisfied himself that such must have been the case.) The fluidextract prepared from this sample was tested immediately after its preparation by Börntrager's test as given above, and was found to give the test only very faintly.

About eight months later Dr. W. S. Hubbard's work on the separation of the common emodin-bearing drugs again brought to the author's mind this question of an emodin-like substance in *Viburnum opulus*—or rather *Acer spicatum*. Hubbard found that ether was more satisfactory than petroleum ether in Börntrager's test since the emodin or emodin-like material is more soluble in the former. He also found that the ether extract from rhubarb, obtained in a process similar to that used for Börntrager's test, when shaken with a saturated solution of ferrous sulphate, imparts a deep blue color to the aqueous solution; also, that when the same ether extract was shaken with calcium hypochlorite solution a red color developed in the aqueous solution.

The fluidextract of *Acer spicatum* (supposed *Viburnum opulus*) which had been prepared eight months before, was examined by these tests in the following manner:

The diluted, acidified fluidextract was shaken with one eighth of its volume of ether (not petroleum ether) and the ether layer decanted off and used for the tests. When underlaid with 10 per cent. ammonia water a brilliant red ring appeared at once. When shaken with a saturated solution of ferrous sulphate the same deep blue color found by Hubbard with rhubarb extracts appeared in the aqueous layer. However, when shaken with calcium hypochlorite solution, only a faint yellow color appeared in the aqueous layer. The fluidextract itself appeared to have become darker in color on standing. These tests were also tried on several extracts of *Viburnum prunifolium* with negative results, only a slight yellow color being imparted to the aqueous solution in any of the tests.

Some months later the author entered the Laboratory of the American Medical Association. There were available samples of fluidextracts of *Acer spicatum* and *Viburnum opulus* prepared by Mr. L. E. Warren from drugs which had been identified by a pharmacognocist. The fluidextract of *Acer spicatum* was about four months old when tested, that of *Viburnum opulus* thirteen months. These were subjected to the above tests. The fluidextract of *Acer spicatum* gave the tests described very positively, just as the fluidextract

which the author had prepared had done. That of *Viburnum opulus* gave results exactly similar to those previously obtained with fluidextracts of *Viburnum prunifolium*. The author was thus led to believe that the sample from which the fluidextract on which he had worked previously had been prepared was in fact *Acer spicatum*. The report of the Bureau's pharmacognocist later bore out this fact.

A fresh fluidextract of *Acer spicatum* was prepared from the standardized material available. Immediately after preparation it was found to give the tests with ammonia water and ferrous sulphate solution described above, but, as had been noted before, the color obtained in the test with ammonia water is not as intense as that obtained with older extracts. This apparent increase, on aging, in the amount of the material which gives the test with ammonia, as indicated by the intensity of this test, opens the question: is it not present originally as a different compound which is changed either by simple oxidation or by means of ferment existing with it in the bark, into the substance which gives the reaction?

Farwell (*Bull. of Pharmacy*, 1913, XXXIII, p. 65) points out that most of the drug sold as *Viburnum opulus* is in fact *Acer spicatum*. Kraemer in the second edition of his pharmacognosy also states that the drug described in the U. S. P. VIII is in fact *Acer spicatum*. This accounts for the fact that several commercial fluidextracts of *Viburnum opulus*, as well as a few proprietary remedies claiming to contain *Viburnum opulus*, gave the tests described above.

#### SUMMARY

*Acer spicatum* contains a substance which gives a crimson color with ammonia, and which may be similar to the emodins of the common cathartic drugs. It also contains a substance which gives a blue color with ferrous sulphate solution similar to that obtained from rhubarb.

Further, it seems that these two reactions should be of value for the identification of extract of *Acer spicatum* in medicinal preparations. Hubbard has shown that rhubarb, alone of all the common "emodin-bearing" cathartics, gives the blue color with ferrous sulphate solution. Rhubarb is distinguished from *Acer spicatum* by the red color which the former gives with the calcium hypochlorite test. The identification of *Acer spicatum* in the presence of rhubarb, of course, cannot be accomplished by these tests.

## WHEELER'S TISSUE PHOSPHATES

L. E. Warren, Ph.C., B.S.

(Reprinted, with additions, from *The Journal A. M. A.*,  
May 5, 1917, p. 1337)

"Wheeler's Tissue Phosphates," known also as "Compound Elixir of Phosphates and Calisaya," is advertised as a nerve food and a nutritive tonic. The label states that it contains calcium, iron, sodium trihydrogen phosphates, alkaloids of Peruvian bark with 12½ per cent. of alcohol. The preparation is sold by the T. B. Wheeler, M. D. Co., of Rouses Point, New York. According to the manufacturer, Wheeler's Tissue Phosphates

" . . . is an *inorganic* combination of the phosphates of iron and calcium and hydrogen (phosphoric acid) together with hydrochloric acid, hydrocyanic acid, and quinine, cheerful coloring, and a delicious, coral-like flavoring."

" . . . The iron is the green, inorganic phosphate and the calcium the simple white phosphate of your early student days. . . ."

The preparation is a red liquid, having an acid reaction, a sweet-bitter taste and the odor of wild cherry. Qualitative tests indicated the presence of calcium, iron, a phosphate, a chlorid, a sulphate, quinin or cinchona alkaloids, alcohol, sodium, cochineal coloring and invert sugar. Ammonium salts, glycerol, citrates or lactates were not found. From the quantitative values obtained the preparation may be taken to represent:

Sp. gr. at 25C./25C. ....	1.1087
Alcohol (per cent. by volume) ....	11.35
	Gm. per 100 c.c.
Calcium phosphate $[Ca_3(PO_4)_2]^*$ ....	0.397
Iron phosphate $(FePO_4 \cdot 4H_2O)^*$ ....	0.068
Chlorid (as hydrochloric acid) ....	0.407
Sodium sulphate $(Na_2SO_4 \cdot 10H_2O)$ ....	0.043
Quinin sulphate (U. S. P.) ....	0.041
Sodium phosphate $(Na_2HPO_4 \cdot 12H_2O)$ ....	0.065
Invert sugar ....	26.82
Water, cochineal and flavor to make....	100 c.c.

\* It should be understood that the calcium and iron salts are held in solution by the hydrochloric acid.

The dose of Wheeler's Tissue Phosphates recommended by the manufacturer is a tablespoonful or about 15 c.c. ( $\frac{1}{2}$  oz.). The total calcium in a dose of the preparation is equivalent to about one-sixth of an average dose of the official calcium chlorid, and the total phosphate to each dose is equivalent to about one-fourth of a dose of the official diluted phosphoric acid. Each prescribed dose of the preparation contains about 0.01 gm. ( $\frac{2}{13}$  grain) of iron phosphate or about one twenty-fifth of the average dose, and to obtain a Pharmacopeial dose of iron phosphate the patient would be obliged to take three-fourths of the contents of an entire bottle—or 12 ounces—of the preparation. If it be assumed that all of the chlorid present is in the form of free hydrochloric acid, each dose of the preparation contains the equivalent of about two-thirds of one Pharmacopeial dose of diluted hydrochloric acid. Each dose of the preparation contains about 0.0062 gm. ( $\frac{1}{10}$  grain) of quinin sulphate, or about one-sixteenth of the average tonic dose. In other words, to obtain the amount of quinin sulphate given in the U. S. Pharmacopeia as the tonic dose, the patient would be required to swallow  $7\frac{1}{2}$  fluidounces of the proprietary preparation, or the contents of nearly half a bottle. The fallacy of prescribing Wheeler's Tissue Phosphates either for its quinin or its iron content is apparent.

Wheeler's Tissue Phosphates is, then, a mildly bitter, flavored syrup which contains nearly 12 per cent. of alcohol, small quantities each of calcium phosphate and hydrochloric acid and insignificant amounts of iron and quinin salts. In other words, essentially it is a sweetened solution of small quantities of calcium phosphate in very dilute hydrochlorid acid together with 12 per cent. of alcohol.

Bearing in mind the analysis of the preparation, how ludicrous some of the claims appear:

...“*Tissue Phosphates* is not a hypophosphate preparation; it is not a combination of glycerophosphates or other organic salts, or so called peptonates and manganates, all recently condemned by the best therapeutic opinion here and in Europe, as much slower and less active than the simpler salts. The iron is the green, inorganic phosphate and the calcium the simple white phosphate of your early student days. Nature takes these simple salts and builds them rapidly into lecithin, bone, and other tissue, without the delay incurred by splitting up the organic salts before she can recombine them.”

“*Tissue phosphates* is in fact a *chemical food*.”

“The formula, suggested by Professor Dusart, of Paris, combines in an easily assimilable and agreeable cordial; medium medicinal doses of Phosphorus, the Generator of Nerve Force; Calcium Phosphate, for

Cell Development and Nutrition; Sodium Phosphate, a stimulant of Liver and Pancreas and Corrective of Acid Fermentation in the Alimentary Canal; Iron, generating in the Blood Heat and Motion; Phosphoric Acid, Tonic in Sexual Debility; Alkaloids of Calisaya, Antimalarial and Antipyretic; Extract of Wild Cherry, Tonic, yet Calming Irritation and Diminishing Nervous Excitement; Ethyl Alcohol 12.5%; and Aromatics."

Although the claim is made that the "formula" of Wheeler's Tissue Phosphates had been "suggested by Professor Dusart," such of Dusart's papers as were available in this country<sup>1</sup> failed to disclose any "formula" that was at all comparable to this product.

Concerning the preparation and the methods by which it is exploited *The Journal* commented as follows:

The investigation verifies facts that must be obvious to every physician who has given the matter thought. "Wheeler's Tissue Phosphates" is an unscientific, shotgun mixture whose most active and powerful drug is the alcohol it contains. That it was not years ago relegated to the realms of obsolete and discarded preparations is a commentary alike on the lack of scientific discrimination and the persuasive power of advertising. While in the past "Wheeler's Tissue Phosphates" has been advertised extensively in medical journals, it seems that now the chief, if not the only, beneficiary of the advertising appropriation for this product is the *New York Medical Journal*, which weekly heralds the "Delicious" and "Sustaining" qualities of "The Ideal Tonic for Fastidious Convalescents."

The following notes on the history of the phosphates (and the hypophosphites) as medicinal substances were compiled in the laboratory:

The manufacturers of Wheeler's Tissue Phosphates, as before stated, have claimed that the formula for their preparation was suggested by Prof. Dusart of Paris. As no similar formula could be found in Prof. Dusart's available papers it seemed worth while to prepare a brief abstract of his experiments and conclusions.

Apparently, Dusart's phosphate preparation was made by treating calcium phosphate with diluted lactic acid, filtering

1. Dusart, L.: *Recherches expérimentales sur le rôle physiologique et thérapeutique du phosphate de chaux*, Paris, 1870; *Quel est l'acide du suc gastrique?* Lille, 1874, unbound, 8 pages; *Notice sur l'emploi et les propriétés du lacto-phosphate de chaux*, Clichy, 1868, unbound, 8 pages. Dusart and Blache: *Recherches sur l'assimilation du phosphate de chaux*, Paris, 1868, unbound, 15 pages.

and adding syrup to the filtrate. The presence of iron, phosphoric acid or cinchona alkaloids was not mentioned. Dr. Dusart's papers were examined with the object of determining, if possible, how he first came to use the phosphates in therapy. It appears that he believed lactic acid to be the chief cause of the acidity of the gastric juice. His experiments showed that commercial calcium phosphate was rendered partially soluble by treatment with dilute lactic acid (0.25 per cent.) and also by the gastric juice of dogs. The solution of calcium phosphate in lactic acid he called "lacto-phosphate of lime." Dusart fed this to guinea-pigs suffering from fractures and, in addition to the usual diet of carrots, used as controls animals under the same diet and injury but without calcium lactophosphate. From experiments on six test animals with four controls he concluded that the bones of animals unite more quickly by the administration of calcium lactophosphate. He claimed to find it of value also in the treatment of rickets. He came to believe, therefore, that deficiencies of bone substance could be supplied by feeding soluble calcium phosphate. The lactophosphate of calcium was prepared in the form of a syrup, and sold in the pharmacies of Paris. Dusart wrote several brochures extolling the preparation and giving information as to where it could be obtained. In other words, the preparation became a proprietary medicine exploited by Dusart.

It is probable that the therapeutic use of the phosphates in this country began with their advocacy by Dr. Samuel Jackson of Philadelphia. He used the phosphates in rickets, probably as a disciple of the teachings of Dusart. As a result of Jackson's theories concerning malnutrition "Jackson's Bone Food," "Chemical Food," the "Compound Syrup of the Phosphates," came into vogue. The "Compound Syrup of Phosphates" in name, appearance and use so closely resembled the "Compound Syrup of Hypophosphites" that much confusion arose in the lay mind as to the distinction between the two preparations. To prevent or overcome this, pharmacists colored the phosphate preparation, usually with cochineal, and flavored it, commonly with spearmint or wild cherry. Few of the phosphate preparations became popular, probably because they were not advertised extensively, and only one proprietary preparation, "Wheeler's Tissue Phosphates," appears to have survived to the present. That this has been kept alive is probably due to persistent advertising

by the promoters and the lack of discrimination in prescribing by physicians.

The use of the phosphates in medicine began a little later (1868) than that of the hypophosphites (1858), and may have been suggested to Dusart by the popularity of the salts last named. The hypophosphites were first exploited by Dr. John F. Churchill<sup>2</sup> for the cure of consumption in the belief that phosphorus deficiency is the cause of tuberculosis, and that the hypophosphites were capable of supplying the lacking element. The untenability of the theory was fully discussed in *The Journal* several years ago,<sup>3</sup> and the uselessness of the hypophosphites as sources of phosphorus for the body has been pointed out frequently.<sup>4</sup>

### Details of Analysis

*Alcohol.*—Alcohol was determined by the provisional method of the Association of Official Agricultural Chemists (Bur. of Chem. Bull., 107, p. 83). The specific gravity of the distillate from 100 c.c. of the material at 15.6 C. was found to be 0.98507 equivalent to 11.35 per cent. of alcohol by volume.

*Residue on Drying in Vacuum.*—A weighed amount of the material was dried under reduced pressure at the temperature of boiling water. The material charred so that no attempt was made to complete the experiment.

*Calcium.*—A measured quantity of the material was made alkaline with ammonium hydroxid and sufficient citric acid solution added to dissolve the precipitate formed. An excess of ammonium oxalate solution was added, the precipitate collected in a weighed Gooch crucible, washed, dried, heated gently and weighed as calcium carbonate. From 100 c.c. of the material 0.3837 gm. of calcium carbonate was obtained, equivalent to 0.1537 gm. of calcium. A duplicate of 25 c.c. gave 0.0762 gm. of calcium carbonate, equivalent to 0.1541 gm.

2. Churchill, J. F.: *De la cause immédiate et du traitement spécifique de la phthisie pulmonaire et des maladies tuberculeuses*, Paris, 1858.  
Churchill, J. F.: *Du moyen de prévenir la phthisie par l'emploi des hypophites*. Paris, 1859.

3. The Hypophosphate Fallacy: *THE JOURNAL*, April 25, 1914, p. 1346; Sept. 2, 1916, p. 760.

4. The Fallacy of Hypophosphate Treatment, *THE JOURNAL*, March 8, 1913, p. 747.

Marriott, W. McKim: The Therapeutic Value of the Hypophosphites, *THE JOURNAL*, Feb. 12, 1916, p. 486.

Compound Syrup of Hypophosphites, *THE JOURNAL*, April 8, 1916, p. 1068.

of calcium per 100 c.c. Average: 0.1539 gm. of calcium per 100 c.c.

*Phosphoric Acid.*—This was determined in a measured quantity of the dealecoholized preparation by precipitation with ammonium molybdate, solution of the precipitate in ammonia water, precipitation with magnesia mixture and weighing the ignited precipitate as magnesium pyrophosphate in the usual way. From 100 c.c. of the material 0.3418 gm. of magnesium pyrophosphate was obtained, equivalent to 0.3009 gm. of phosphoric acid ( $H_3PO_4$ ). A duplicate of 20 c.c. gave 0.0696 gm. of magnesium pyrophosphate, equivalent to 0.3063 gm. of phosphoric acid per 100 c.c. Average: 0.3036 gm. of phosphoric acid per 100 c.c.

*Iron.*—The filtrate from the calcium determination was made alkaline with ammonia water, evaporated to dryness, the residue ignited to expel ammonium salts and destroy citrates, the residue digested with hydrochloric acid, the solution treated with potassium iodid solution, and the liberated iodin titrated with tenth-normal sodium thiosulphate. The iodin liberated by the iron from 100 c.c. of the material required 3.05 c.c. of tenth-normal sodium thiosulphate, equivalent to 0.0170 gm. of iron. This is equivalent to 0.068 gm. of iron phosphate ( $FePO_4 \cdot 4H_2O$ ) per 100 c.c.

*Quinin.*—The total alkaloids in a measured quantity of the solution were determined by shaking the slightly alkaline solution with chloroform, shaking the chloroform solution with acid, making the acid solution alkaline, and again shaking out with chloroform. The chloroform extract was dried at 120 C. and the residue weighed in the usual way. From 100 c.c. of the preparation 0.0302 gm. of alkaloid was obtained. This is equivalent to 0.0406 gm. of quinin sulphate, U. S. P., per 100 c.c. of the preparation.

*Sulphate.*—The sulphate in a measured quantity of the material was precipitated with barium nitrate solution, the precipitate of barium sulphate collected, heated and weighed in the usual way. The barium sulphate obtained from 50 c.c. of the material weighed 0.0210 gm., equivalent to 0.0176 gm. of sulphuric acid per 100 c.c.

*Chlorid.*—The filtrate from the sulphate determination was diluted to 500 c.c. with water, and the chlorid determined in aliquot portions of 100 c.c. each of this solution by precipitation with silver nitrate. The washed silver chlorid was dis-

solved in strong ammonia water and reprecipitated with nitric acid and a few drops of silver nitrate solution. The precipitate was then collected, heated and weighed in the usual way. From 100 c.c. of the dilution, representing 10 c.c. of original material, 0.1598 gm. of silver chlorid was obtained, equivalent to 0.4062 gm. of hydrochloric acid per 100 c.c. A duplicate gave 0.1607 gm. of silver chlorid, equivalent to 0.4086 gm. of hydrochloric acid per 100 c.c. Average 0.4074 gm. of hydrochloric acid per 100 c.c.

In calculating the proportions of the several ingredients in the preparation from the analytical values found, the order given below was carried out:

The iron was determined, and the results calculated to iron phosphate  $\text{FePO}_4 \cdot 4\text{H}_2\text{O}$ . The phosphoric acid in the iron phosphate was calculated and the result was subtracted from the total phosphoric acid found. The calcium was calculated to calcium phosphate  $\text{Ca}_3(\text{PO}_4)_2$ . The phosphoric acid in this compound was calculated and the result subtracted from the phosphoric acid remaining after that in the iron phosphate had been subtracted from the total phosphoric acid. The balance of the phosphoric acid was then calculated to sodium phosphate  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ . Quinin was calculated to quinin sulphate, U. S. P., and the sulphuric acid in the compound calculated and subtracted from the total sulphuric acid found. The balance of the sulphuric acid was calculated to sodium sulphate.

$$0.0681 \text{ gm. of } \text{FePO}_4 \cdot 4\text{H}_2\text{O} = 0.0299 \text{ gm. of } \text{H}_3\text{PO}_4.$$

$$0.3036 \text{ gm. of } \text{H}_3\text{PO}_4 - 0.0299 \text{ gm. of } \text{H}_3\text{PO}_4 = 0.2737 \text{ gm. of } \text{H}_3\text{PO}_4.$$

$$0.1539 \text{ gm. of Ca} = 0.3973 \text{ gm. of } \text{Ca}_3(\text{PO}_4)_2 = 0.2524 \text{ gm. of } \text{H}_3\text{PO}_4 \text{ combined with Ca.}$$

$$0.2737 \text{ gm. of } \text{H}_3\text{PO}_4 - 0.2524 \text{ gm. of } \text{H}_3\text{PO}_4 = 0.0212 \text{ gm. of } \text{H}_3\text{PO}_4 \text{ remaining to combine with sodium as } \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}.$$

$$0.0212 \text{ gm. of } \text{H}_3\text{PO}_4 = 0.0652 \text{ gm. of } \text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}.$$

$$0.0412 \text{ gm. of quinin sulphate} = 0.0045 \text{ gm. of } \text{H}_2\text{SO}_4.$$

$$0.0176 \text{ gm. of } \text{H}_2\text{SO}_4 - 0.0045 \text{ gm. of } \text{H}_2\text{SO}_4 = 0.0131 \text{ gm. of } \text{H}_2\text{SO}_4.$$

$$0.0130 \text{ gm. of } \text{H}_2\text{SO}_4 = 0.0429 \text{ gm. of } \text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}.$$

*Invert Sugar.*—A portion of the material (25 c.c.) was diluted with about 200 c.c. of water, the solution treated with a slight excess of basic lead acetate solution, the mixture

filtered, the filtrate made slightly alkaline with sodium carbonate solution, the mixture made up to 1,000 c.c. and allowed to stand for several hours. The supernatant liquid was decanted through a filter, and portions of the filtered solution used for qualitative and quantitative tests for sugars. To 50 c.c. of the clarified solution 1 c.c. of a 1 per cent. aqueous solution of methylene blue was added and the mixture heated. The solution was decolorized before the boiling point was reached. The invert sugar was determined before inversion by the Munson and Walker method (*Bur. Chem. Bull.*, 107, p. 241). From 25 c.c. of the clarified solution, representing 0.625 c.c. of the original material, 0.3604 gm. of cuprous oxid was obtained, equivalent to 0.1677 gm. of invert sugar, or 26.832 gm. per 100 c.c. of original material. A duplicate gave 0.3601 gm. of cuprous oxid, equivalent to 0.1676 gm. of invert sugar, or 26.81 gm. per 100 c.c. Average: 26.824 gm. of invert sugar per 100 c.c. of original material. Since sucrose is inverted quantitatively by long standing in presence of free mineral acids, and this specimen of Wheeler's Tissue Phosphates was known to have been manufactured at least two months before the sugar determinations were made, no tests for sucrose were carried out.

*Cochineal*.—Cochineal was detected by the method formerly used in this laboratory for the detection of that coloring substance in Papine (*Rep. Lab. Am. Med. Assoc.*, 1911, IV, 84). Briefly the method is as follows:

A portion of the dealeoholized specimen was slightly acidified with acetic acid, the mixture shaken with amyl-alcohol, the solvent washed with water and shaken with 1 per cent. aqueous solution of uranium nitrate. A deep green color was produced at once.

### "AMBRINE" AND PARAFFIN FILMS

Paul Nicholas Leech, Ph.D.

(*Reprinted from The Journal A. M. A.*, May 19, 1917, p. 1197)

In the last year or so, the hot-wax or paraffin treatment of burns has been widely discussed both in medical and lay periodicals. Although the treatment is simply a modification of the well-known use of oil and ointments, it has received unusual attention, owing to the widespread sensationalism

following the exploitation in France of a secret and therefore mysterious mixture, "Ambrine," the formula of Dr. Barthe de Sandfort. Owing to this publicity, it seemed desirable to investigate the chemical composition, and to compare its physical properties with other waxlike substances.

"Ambrine" is promoted as a dressing for burns, frostbites, neuritis, varicose ulcers, phlebitis, neuralgia, rheumatism, sciatica, gout, etc. It is a smoky-appearing substance, resembling paraffin in consistency and without odor. For

zed, owing to  
r medium.

*HYPERTHERMALITY A REALITY.*

Hyperthermality is a fact, however, through the agency of a keri-resinous product which has been used in France since 1900 under the name of L'Ambrine. Hyperthermine, as the remedial agent will be known in this country, is a combination of several kinds of waxes and resins, scientifically blended and containing no medicinal elements whatever. It comes in the form of waxy flakes. It melts at 124° and on cooling resembles a dark colored wax.

Hyperthermine is the discovery of Dr. Barthe de Sandfort, an eminent retired French naval surgeon and a member of numerous foreign medical societies. He

"Ambrine" has been exploited in the United States for some time. To physicians it was sold under the name "Hyperthermine." Above is a photographic reproduction (reduced) of a portion of a booklet describing "Hyperthermine," which has been in THE JOURNAL office for some years.

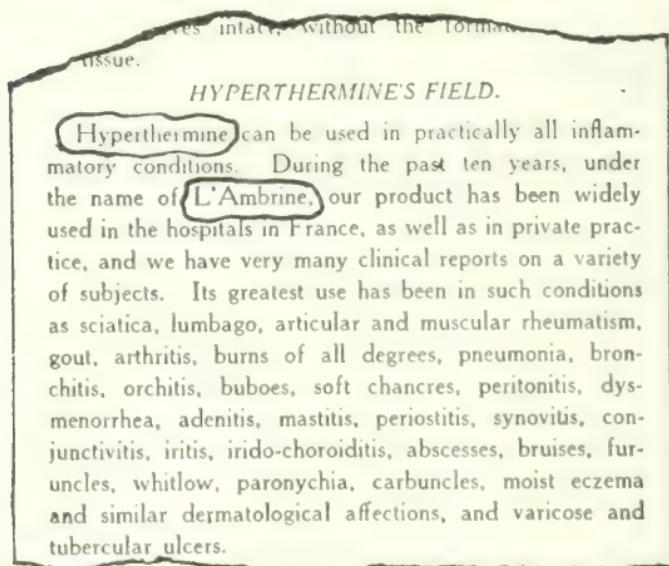
application, "Ambrine" is melted and applied to the wound either with a brush or with a specially devised atomizer. It cools quickly, and leaves a solid, protecting film.

It is said that de Sandfort "stumbled on this treatment by accident."<sup>1</sup> Being a sufferer of rheumatism, he had been benefited by hot mud baths; on returning home he sought a substitute, and finally made a mixture of paraffin, oil of amber and amber resin. This was applied hot, serving as a firm poultice. "Years later, he went on service to a railway in

1. The Outlook, Jan. 17, 1917, p. 100.

China and was in Yunnan at the time of the incendiary insurrection, and many badly burned Chinese were brought in for treatment. Remembering that Ambroise Paré treated such cases with hot oil, he tried the effect of covering the burn with his melted ambrine which at once glazes over, forming a coat impervious to the air, and his patients ceased to suffer."<sup>2</sup>

"Ambrine" has been sold in America under two names: "Hyperthermine," as exploited to physicians, and "Thermo-



Photographic reproduction (reduced) from the "Hyperthermine" ("Ambrine") booklet recommending it for use in rheumatism, gout, pneumonia, buboes, dysmenorrhea, eczema, tuberculous ulcers, etc.

zine," as advertised to the public. Physical comparison alone shows that Ambrine as now sold differs from "Hyperthermine" of a few years ago; the probable reason is that "Ambrine" has changed its formula. This is borne out by Matas,<sup>3</sup> who states that de Sandfort "admitted that Ambrine was a compound of paraffin, oil of sesame and resins, but was not at liberty to divulge its exact composition, as the formula and manufacture of this substance was now the

2. Med. Rec., New York, Jan. 27, 1917, p. 160.

3. Matas, Rudolph: Burns Treated with Paraffin Mixtures, New Orleans Med. and Surg. Jour., April, 1917, p. 681.

property of a private corporation which was exploiting it as a proprietary and secret remedy." The later formula differs from the original.

Besides the foregoing paraffin preparations, two others have recently been placed on the American market, "Parresine" (nonsecret) and "Mulene" (secret).

#### ANALYSIS OF AMBRINE

"Ambrane" comes in rectangular cakes, about  $1\frac{1}{2}$  inches wide, 6 inches long and  $\frac{1}{2}$  inch thick. It is moderately soft, but somewhat brittle at ordinary room temperature. A black substance is present which evidently settles out during the compounding, as in one side of the cake these particles can be clearly discerned by holding it up to the light; in the other side there are no suspended particles. When melted, the solution is not clear, and a sediment forms. The melting point (U. S. P. method; see later) is 48.4 C. The plasticity and ductility<sup>4</sup> are 27 and 30.5, respectively. It is pliable and strong at body temperature. The saponification number and acid number are both very low, but a fatty oil is present. Tests indicated oil of sesame. Ninety-eight per cent. of "Ambrane" is soluble in ether; this soluble portion may be treated with low-boiling ligroin (petroleum ether), out of which, on standing, a black asphalt-like substance separates. Of the ether-insoluble substance, 65 per cent. is soluble in chloroform. The remaining insoluble substance contains a small amount of silica and vegetable fiber. The paraffin obtained from "Ambrane" melted at 48.6 C. As a result of various experiments, it appears that the composition of "Ambrane" is essentially as follows:

Paraffin (M. P. 48.6 C.) .....	97.0 per cent.
Fatty oil (sesame?) .....	1.5 per cent.
Asphalt-like body .....	0.5 per cent.
Coloring matter, and undetermined .....	1.0 per cent.
	100.0

#### OTHER PROPRIETARY FILMS

A cursory examination of "Mulene," manufactured by the Mulene Company, Pittsburgh, was also made. This appears to contain paraffin, beeswax, a fat-soluble red dye and considerable rosin. When heated carefully in a beaker, the

4. These determinations will be described later.

rosin "sticks" to the bottom, and does not go into solution readily.<sup>5</sup>

"Parresine," according to the manufacturers, is a mixture composed of paraffin, 94 to 96 per cent.; gum elemi, 0.20 to 0.25 per cent.; Japan wax, 0.40 to 0.50 per cent.; asphalt, 0.20 to 0.25 per cent., and eucalyptol, 2 per cent., the whole being colored with alkannin and gentian violet.<sup>6</sup>

#### FORMULA FOR PARAFFIN FILM

In a recent article, Sollmann<sup>7</sup> presented various suggestions for the compounding of paraffin films. Some of the formulas were promising and others were not, but all were simple. He did not try to imitate "Ambrine." Lieut.-Col. A. J. Hull<sup>8</sup> of the Royal Army Medical Corps, after experimenting with different combinations, concluded that a mixture of "1 part resorein, 2 parts eucalyptus oil, 5 parts olive oil, 25 parts soft paraffin [petrolatum]<sup>9</sup> and 67 parts hard paraffin" served the purpose as well as "Ambrine." The following formula, which might be called Asphalt-Paraffin No. 21, much more closely resembles "Ambrine," and it seems to have certain advantages, due to the use of a more suitable grade of paraffin:

Paraffin <sup>10</sup> (M. P. 44 U. S. P. melting 47.2° C.) . . . . .	97.5 gm.
Asphalt . . . . .	from 3 to 5 drops
Olive oil . . . . .	1.5 c.c.

About 10 c.c. of "asphalt varnish" (B. Asphaltum)<sup>11</sup> is placed in a beaker and heated on the steam bath for one-half hour. From 3 to 5 drops, delivered from a 1 c.c. pipet, are then placed in a casserole, and 1.5 c.c. of olive oil added.

5. When the sample was first obtained, this feature was not observed.

6. Made by the Abbott Laboratories, Chicago, and accepted by the Council on Pharmacy and Chemistry for New and Nonofficial Remedies, THE JOURNAL, May 12, 1917, p. 1406.

7. No chemical examination was made.

8. Sollmann, Torald: Suggested Formulas for Paraffin Films, THE JOURNAL A. M. A., April 7, 1917, p. 1037.

9. Hull, A. J.: The Treatment of Burns by Paraffin, Brit. Med. Jour., Jan. 13, 1917, p. 37; The Treatment of Burns by Paraffin, Therapeutics, THE JOURNAL A. M. A., Feb. 3, 1917, p. 373.

10. The "soft paraffin" of the British Pharmacopeia resembles petrolatum, U. S. P., Queries and Minor Notes, THE JOURNAL A. M. A., April 28, 1917, p. 1281.

11. The paraffin used in this formula was supplied by the Standard Oil Company of Indiana; the melting point given by the manufacturers is from 120 to 122 F. which, according to the American Standard of taking melting points, gives higher results than the method described in the pharmacopeia.

12. The "Asphalt Varnish" used was obtained from Remien and Kishner Company, Chicago.

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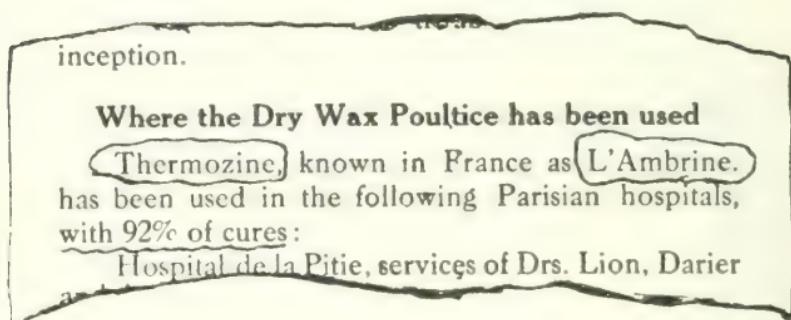
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Photographic reproduction (greatly reduced) of a full page magazine advertisement of "Thermozone," the name under which "Ambrine" was sold to the public.

The mixture is heated and stirred for a few minutes until perfect solution is effected. To this is then added, with stirring, the paraffin, which has been previously melted. When it is cooled, a brown solid is obtained.<sup>13</sup> The physical factors of this paraffin mixture are, melting point 45.4 C. (U. S. P. method); plasticity, 28.5; ductility, 29; it is very pliable and strong at 38 C., and adheres exceedingly well to the skin, although it detaches easily. This mixture, which is easy to prepare, is inexpensive, the cost of the materials being approximately 10 cents a pound.

Both Hull and Sollmann noticed that tarlike substances and melted paraffin do not mix well. This is noticeable in "Ambrane," which cannot be called an "elegant" preparation.



Photographic reproduction from a booklet on "Thermozine" showing that it is identical with "Ambrane."

The difficulty may be overcome by first mixing hot olive oil and asphalt; the asphalt will then go into solution. It is interesting to note that the suggested formula (as well as others which were also prepared) is not as plastic as the paraffin itself.<sup>14</sup> This is also true of "Ambrane." On the other hand, the melting point of the paraffin is higher. *The important point, however, in compounding all paraffin preparations, is to select a proper grade of paraffin as elaborated below.*

13. While needless, a color resembling "Ambrane" may be obtained by the addition of coloring agents.

14. In a personal communication Dr. Sollmann expressed the opinion that the synthetic preparation is inferior to the paraffin used in the formula, basing the view on the greater plasticity of the paraffin. For practical purposes, the paraffin will most probably serve as well as the mixture, especially when it is held in place by bandages, but I believe that the mixture is more adhesive.

EXAMINATION OF PARAFFINS AND PARAFFIN PREPARATIONS

The name "paraffin" generally applies to a colorless and tasteless waxlike substance that is solid at ordinary temperature. It is composed of saturated hydrocarbons, that is, they are unable to take up any more hydrogen, and thereby are quite stable; the hydrocarbons in paraffin have the general formula of  $C_nH_{2n+2}$ , ranging as high as  $C_{24}H_{50}$  to  $C_{27}H_{56}$ . Paraffin may be found in crude form in coal, from which source the first paraffin candles were made. It may be produced from the distillation of brown coal, as in Germany, or from bituminous shale. In America, it is

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Bronchitis	Felons	Neuralgia	Synovitis
Bruises	Gout	Open Sores	Tonsilitis
Burns	Laryngitis	Pneumonia	Ulcers

and any and all inflammations and swellings.

These conditions show a partial list of the indications for Thermozine's

Photographic reproduction from a booklet on "Thermozine" giving the conditions in which the stuff was alleged to be "very useful."

obtained chiefly from the distillation of crude petroleum, being in the residue after the distillation of such products as naphtha (gasoline), kerosene and the lubricating oils. The residue is treated by one of a number of processes causing the unpurified solid paraffin to be made available. The crude paraffin is either sold as such, or is refined. Paraffin or "paraffin waxes"<sup>15</sup> are designated in the trade by their melting points (which in the "American standard" is

15. Paraffin is sometimes spoken of as "white wax." This is unfortunate, as "white wax" is an official name for "White Beeswax, U. S. P." The term "white wax" is also often applied to "Chinese wax," which is formed from an insect living on the tree *Ligustrum lucidum*.

expressed in Fahrenheit degrees), and as to their state of refinement as "crude," "semirefined" and "fully refined" paraffin. There are certain chemical and physical differences so that two refined waxes having the same melting point would not have the same plasticity. The higher melting point varieties of paraffin are hard and tough at room temperature; when melted, paraffin expands and forms a thin mobile liquid.

The significant requirements of paraffin for surgical dressings are that it should be solid at body temperature, at the same time having flexibility and adhesiveness, together with a certain amount of strength. A number of brands of paraffin are sold in the United States, so that it seemed advisable to examine some of them and compare them with certain paraffin-film preparations. They were tested as to their melting points, plasticity, ductility, strength of film, etc.

*Melting Point Determination.*—The melting point was determined by the method of the U. S. Pharmacopeia IX, p. 596. The melting point as obtained by this method is lower than the melting point used by manufacturers of paraffin (after conversion to Fahrenheit).

*Pliability and Ductility, Limit Temperature.<sup>16</sup>*—A little of the melted wax was poured from a teaspoon on the surface of the water at about 40°C., in a tin pan (bread mold). This formed a fairly thin film. The temperature of the water was then lowered by the addition of cold water. At each temperature the pliability and ductility were tested thus:

*Pliability Test.*—The film, immersed in water, was doubled on itself, note being taken whether or not it broke.

*Ductility Test.*—The film was pulled under water, note being taken whether it stretched on being pulled and broke with a ragged fracture; or whether it broke sharp without stretching. It is desirable that the pliability and ductility be preserved at as low a temperature as possible.

*Cotton Films, Adhesives and Detachability.<sup>17</sup>*—The melted wax was applied as it would be for burns; namely, a thin layer was painted on the inner surface of the forearm with a camel's hair brush,<sup>17</sup> a transverse strip about an inch wide being made. This was covered with a very thin layer of absorbent cotton, and over this another layer of melted wax

<sup>16</sup> I am indebted to Dr. Terrell Sollmann for these methods.

<sup>17</sup> When painting a surface with a paraffin film, I found that the temperature of the paraffin should not be too close to the melting point, but several degrees above; otherwise it does not "set" well.

was painted. As soon as this had cooled a little, it was covered by a few layers of bandage and left on for at least an hour. At the end of that time, the bandage was removed. The cotton film should be found at the place at which it was applied, showing that it is sufficiently adherent. It should detach without "pulling" the skin.

The results of these tests are given in the accompanying table. It can be seen that nearly all the paraffins examined have properties which would make them useful, the notable exceptions being Nos. 8, 15 and 16. The more satisfactory



Photographic reproduction (greatly reduced) of the carton in which "Ambrane" is now sold.

products would be those having a melting point about 47 C., ductility of 39 or below, and plasticity of 28 or below. The paraffin described in the U. S. Pharmacopeia is not so satisfactory, the required melting point being between 50 and 57 C.

The use of paraffin bandages has been suggested by Fisher and Sollmann.<sup>18</sup> In such cases, it may very likely be that a paraffin of higher melting point would be more satisfactory, owing to its greater resistance and tougher fiber.

18. Fisher, H. E.: Nonadhering Surgical Gauze, THE JOURNAL A. M. A., March 25, 1916, p. 939.

19. Sollmann, Torald: Paraffin-Covered Bandages, THE JOURNAL A. M. A., April 21, 1917, p. 1178.

PARAFFINS AND PARAFFIN PREPARATIONS. TABLE A

Formula No.	Substance	Melting Point, U. S. P.	Ductility Limit	Plasticity Limit	(a) Adhesiveness and Detachability (b) Strength of Film at 38° C.
1	"Parowax," Stand. Oil Co. of Ind. ....	50.8	32.5	29.0	(a) Adheres and detaches well; rather hard (b) Pliable and strong
2	"Paraffin 118-120 F," Stand. Oil Co. of Ind. ....	46.8	28.5	24.5	(a) Does not adhere well; detaches easily (b) Pliable but not strong
4	"Paraffin 126-122 F," Stand. Oil Co. of Ind. ....	47.2	25.0	24.5	(a) Adheres well; detaches well (b) Pliable and fairly strong
5	"Paraffin 126-125 F," Stand. Oil Co. of Ind. ....	48.8	31.5	28.5	Same as 4
6	"Paraffin 128-120 F," Stand. Oil Co. of Ind. ....	52.0	33.0	30.0	(a) Adheres well; detaches not so easily (b) Pliable and strong
7	"Texwax," Texas Co., Port Arthur, Texas, ....	51.2	32.5	29.8	Same as 6
8	"Paraffin Wax 126-124 F," Warren Refining Co., Warren, Pa.	50.6	36.0	34.5	(a) Unsatisfactory; does not adhere (b) Only slightly pliable; too tough
9	"Paraffin No. 910," Waverly Oil Works, Pittsburgh.	47.0	30.5	26-27	(a) Adheres well; detaches well (b) Pliable and strong
10	"Paraffin No. 929," Waverly Oil Works, Pittsburgh.	41.4	27.5	25.0	(a) Adheres well; detaches well (b) Pliable and fairly strong
11	"Hard Paraffin," Robt Stevenson & Co., Chicago.	48.0	28.5	24.5-25.5	(a) Adheres well; detaches well (b) Pliable and strong
12	"Paraffin," Island Petroleum Co., Chicago, ....	47.2	33.0	32.5	Not quite as good as 11
13	"Paraffin 122 F," Gulf Refining Co., Pittsburgh, ....	46.8	30.5	27.5-28	(a) Does not adhere so well; detaches well (b) Very pliable
14	"Paraffin 125 F," Gulf Refining Co., Pittsburgh, ....	50.0	32.0	31.0	About as 13
15	"Paraffin 132 F," Gulf Refining Co., Pittsburgh, ....	54.8	35.5	34.0	(a) Does not adhere well (b) Not very pliable, but strong

16	"Paraffin No. 301," National Refining Co., Cleveland.:	50.2	33.0	32-32.5	(a) Does not adhere well (b) Not very pliable
18	Paraffin recovered from "Ambrine".....	48.6	30.5	28-28.5	(a) Adheres well; detaches well (b) Pliable but not strong
19	"Hyperthermine" .....	49.4	33.5	30.5-31	(a) Does not adhere well; detaches well (b) Very pliable and strong
20	"Ambrane" .....	48.4	30.5	27.0	(a) Adheres well; detaches well (b) Very pliable and strong
21	Paraffin 120-122 F. (see 4), 97.5; olive oil, 1.5; asphalt, 4 drops	45.4	29.0	28.5	(a) Adheres excellently; detaches well (b) Very pliable and strong
22	"Parowax" (see 1), 97.5; olive oil, 1.5; asphalt, 4 drops	49.2	32.0	30.5	(a) Adheres well; detaches well (b) Pliable and strong
23	"Mulene" .....	51.0	36.0	28.0	(a) Adheres but detaches with difficulty (b) Pliable but not strong
24	"Parresine," Abbott Laboratories, Chicago.....	46.0	26.5	26.0	(a) Adheres well; detaches easily (b) Pliable and fairly strong
25	"Paraffin 118-121 F," The Atlantic Refining Co., Philadelphia .....	45.8	26.4	23.2	(a) Adheres well; detaches easily (b) Pliable and fairly strong

TABLE B

Formula No.	Substance	Melting Point, U. S. P.	Ductility Limit	Plasticity Limit	(a) Adhesiveness and Detachability (b) Strength of Film at 38 C.
26	"Cereleene," Holliday Lab., Pittsburgh.....	50.0	30.5	26.5	(a) Adheres well; detaches with pulling (b) Not strong at 38 C.
27	"Stanolinid" Surgical Wax,† Standard Oil Co. of Ind.	47.0	28.8	25.0	(a) Adheres well; detaches easily (b) Fairly strong at 38 C.

\* On being heated, it readily loses eucalyptol, and a small amount of resinous substance forms in the bottom of the beaker. If "Cereleene" is heated to 145 C. and cooled, the resulting product no longer has the properties of the original "Cereleene."  
 † Accepted by the Council on Pharmacy and Chemistry for inclusion in New and Nonofficial Remedies.

## SUMMARY

1. "Ambrine" is essentially paraffin in which a small amount of fatty and asphalt-like body is incorporated; like most secret mixtures, its composition varies.

2. A simple formula for a paraffin film, similar in chemical composition but superior in physical properties to "Ambrine," is that described as Formula 21. The superiority is due to using a grade of paraffin that is better adapted to the purpose. The cost of materials is about 10 cents a pound.

3. The properties of the paraffin used for a surgical dressing are important. A number of different grades have been examined, in order to determine the ones that appear most promising. Paraffins Nos. 3, 4, 10, 11 and 25 are the best in the table, and surpass "Ambrine" itself.

4. It is exceedingly probable that further experience will show that for most purposes simple paraffin will serve just as well as the mixtures—if, indeed, not better.

## Addenda

Since the foregoing was published, two other products—"Cerelene" and "Stanolind Surgical Wax"—were submitted to the Council on Pharmacy and Chemistry for investigation as to their acceptability for inclusion in New and Nonofficial Remedies. In this connection the Laboratory was requested to examine them.

"Cerelene" is manufactured by the Holliday Laboratories, Pittsburgh. According to the manufacturers, "Cerelene" is a compound composed of 84 per cent. paraffin, 15 per cent. myricyl palmitate and 1 per cent. elemi gum. As ordinarily marketed, "Cerelene" contains the following materials: To the beeswax is added Oil of Eucalyptus, U. S. P., 2 per cent. and Betanaphthol, U. S. P., 0.25 per cent. The manufacturer further states that the myricyl palmitate is a purified form of beeswax, free from all impurities, acids, etc., which is solely manufactured by this company and for which patents are pending. The properties described for "Cerelene" were as follows:

When cold, Cerelene is a solid wax-like cake of a tawny yellow brown color. On exposure to air for long periods, the amber color darkens to some extent. It is entirely free from solids, odorless and tasteless; does not separate or change when melted repeatedly, and cannot in the melted state be separated by fractional crystallization. It is entirely neutral to indicators, being perfectly free from both acids and bases.

Tests: Melting Point, U. S. P. method, 126 F.

Density, U. S. P. method, 0.907.

Iodin value, 0.5.

Saponification number, 0.9.

"Stanolind Surgical Wax" is manufactured by the Standard Oil Company of Indiana. In the submission of the product to the Council on Pharmacy and Chemistry, it was stated that the product was a specially prepared paraffin "free from dirt or other deleterious matter. . . . It has been steamed and restamed to drive out any free oil repeatedly filtered."

The examination of the foregoing products yielded the figures described in Table "B."

## THE STABILITY OF IODINE OINTMENTS

L. E. Warren, Ph.C., B.S.

(Reprinted from the *American Journal of Pharmacy*,  
April, 1917, p. 559)

In general, the literature on the keeping qualities of iodine ointment, and on the stability of iodine if mixed with ointment bases, is confusing. The recorded evidence is often contradictory. The attention of the writer was brought to this condition by studies of several proprietary preparations, Iodex,<sup>1</sup> Iod-Izd-Oil,<sup>2</sup> Locamfen,<sup>3</sup> and Locamfen<sup>3</sup> Ointment.

Iodex was sold under the claim that it is

" . . . an embodiment of vaporized iodine, in an organic base, reduced and standardized at 5 per cent. by incorporation with a refined petroleum product."

The exact composition of Iodex is a trade secret. Analysis showed that it contains petrolatum-like substances and combined iodine, the latter probably in combination with oleic acid. Tests for free iodine were made in five specimens of Iodex. In one of these no free iodine was present; in the others the merest traces were found.

Two years ago a preparation called "Iod-Izd-Oil" was examined. This was claimed to contain 2 per cent. of free iodine in liquid petrolatum. At the time of the examination the age of the preparation was not known, but it had been obtained just prior to the analysis, and was thought not to be very old. The analysis showed that it contained but about 0.43 per cent. of iodine, all of which was in a free state. The fact that all of the iodine present was in the free state

1. Rep. Chem. Lab., A. M. A., 1915, 8, 89.

2. Rep. Chem. Lab., A. M. A., 1915, 8, 106.

3. Rep. Chem. Lab., A. M. A., 1916, 9, 118.

appeared to indicate that iodine is relatively stable in liquid petrolatum solutions.

Iocamfen is a liquid composed of iodine, camphor and phenol. It was claimed to contain 10 per cent. of free iodine. Analysis showed that it contained 9.3 per cent. of total iodine (of which 7.5 per cent. was present in an uncombined state), 66.1 per cent. of camphor and 19.7 per cent. of phenol. After storing for several months a second assay of Iocamfen showed no appreciable loss in iodine content. This would indicate that iodine is relatively stable in presence of phenol and camphor, although immediately after mixing there is some loss of free iodine. The Iocamfen Ointment was supposed to contain 50 per cent. of Iocamfen (equivalent to 5 per cent. of free iodine) in a lard-wax-cacao-butter base. The analysis showed that the ointment contained but 0.4 per cent. of free iodine, the balance being in combination. From the results of the examination, and from correspondence with the manufacturers (Schering and Glatz), it became evident that the only plausible explanation for the loss of free iodine in the preparation of Iocamfen Ointment from Iocamfen lay in the combination of the free iodine with the ingredients of the ointment base. It seems likely that the free iodine originally present in Iocamfen for the most part had gradually gone into combination with the fatty substances after the ointment had been prepared.

The literature was then examined to determine the consensus of opinion concerning the stability of iodine in iodine ointment. In the older literature the belief that iodine ointment is unstable appears to be quite general. Such statements as the following are typical:

The ointment should be prepared only when wanted for use, for it undergoes change if kept, losing its deep, orange-brown color, and becoming pale upon its surface.<sup>4</sup>

It is better to prepare it only as it is required for use.<sup>5</sup>

This ointment must not be dispensed unless it has recently been prepared.<sup>6</sup>

In 1909 Lythgoe,<sup>7</sup> of the Massachusetts Board of Health laboratory, reported an examination of four samples of iodine ointment. Three were found to be pure, the fourth was low

4. U. S. Disp., ed. 19, p. 1315.

5. Am. Disp., ed. 2, p. 2022.

6. U. S. Pharmacopeia, IX, p. 481.

7. Rep. Mass. Bd. Health, 1909, 41, 477.

in iodine. Experiments showed that iodine ointment deteriorates rapidly; consequently, no further collections of samples were made.

In 1912 Pullen<sup>8</sup> reported that he had prepared two specimens of iodine ointment according to the British Pharmacopeia, one being from new lard and the other from a specimen of lard at least two years old. Assays for free iodine were carried out immediately after the preparations were made, and at intervals afterward up to four months. The following values were found:

	Sample I Ointment from new lard, per cent.	Sample II Ointment from old lard, per cent.
Iodine introduced .....	4.0	4.0
Iodine found immediately after making	3.95	3.38
Iodine found after twenty-four hours	3.30	3.15
Iodine found on the third day.....	3.18	2.62
Iodine found on the seventh day....	3.15	2.46
Iodine found on the fourteenth day..	3.00	2.45
Iodine found after one month,.....	3.00	2.39
Iodine found after two months.....	2.90	2.31
Iodine found after four months.....	2.92	2.26

Pullen found that the loss in free iodine could be accounted for by the iodine which had gone into combination with the fats of the ointment base.

Pullen also found that if the potassium iodide and glycerin were omitted in the preparation of the ointment, the loss in free iodine was very rapid, the preparation containing practically no free iodine (only  $\frac{1}{2}\%$ ) after a few hours. He concludes that the use of potassium iodide and glycerin is necessary for the preservation of the ointment. He obtained specimens of iodine ointment in drug stores, and assayed them for free iodine. It is to be presumed that the ages of the several specimens were not known. The results are found in the following table:

Specimen No. 1.....	2.74 per cent.
Specimen No. 2.....	2.85 per cent.
Specimen No. 3.....	2.62 per cent.
Specimen No. 4.....	2.48 per cent.
Specimen No. 5.....	2.53 per cent.
Specimen No. 6.....	2.79 per cent.

8. Pharm. Jour., 1912, 89, 610.

Fried<sup>9</sup> prepared iodine ointment according to the U. S. P. VIII formula, and assayed it at intervals. His results are tabulated herewith:

	Per cent.
Iodine introduced .....	4.00
Iodine found immediately after making.....	3.89
Iodine found one hour after making.....	3.51
Iodine found one day after making.....	3.48
Iodine found five days after making.....	3.06
Iodine found ten days after making.....	2.84
Iodine found thirty days after making.....	2.81
Iodine found ninety days after making.....	2.81
Iodine found eight months after making.....	2.81

Iodine ointment has been official in the U. S. Pharmacopeia since 1870. Briefly, the method now used for making the preparation is as follows:

Four gm. of iodine, 4 gm. of potassium iodide and 12 gm. of glycerin are weighed into a tared mortar and the mixture triturated until the iodine and potassium iodide are dissolved and a dark, reddish-brown, syrupy liquid is produced. Eighty gm. of benzoinated lard are then added in small portions and with trituration after each addition. The mass is then triturated until of uniform consistence.<sup>10</sup>

Iodine ointment is officialized also in several foreign pharmacopeias, although the iodine strength of the several preparations is not uniform. The formula in the British Pharmacopeia is exactly like that in the U. S. Pharmacopeia except that pure lard is directed to be used instead of benzoinated lard. Some of the foreign pharmacopeias also specify that the preparation must be freshly prepared when wanted. In the earlier editions the U. S. Pharmacopeia directed the ointment to be prepared by using water as the solvent for the potassium iodide. In the U. S. Pharmacopeia VIII the formula was changed so as to employ glycerin, and that solvent is now official. Water is still prescribed as the potassium iodide solvent by the Pharmacopeias of the Netherlands and of France.

From the examination of the literature it seems probable that iodine ointments which contain petrolatum products only as the ointment bases are apt to be relatively stable, so far as the content of free iodine is concerned. On the other hand, ointments the bases of which contain fats of the unsaturated

9. Pharm. Jour., 1912, 89, 610.

10. The time required to complete the process after the initial portion of lard had been added should be about twenty minutes.

fatty acid series, such as oleic acid, do not satisfactorily preserve the iodine in the free state. In the latter class it seems likely that the iodine enters into combination with the unsaturated fatty acids. Accordingly, on theoretical grounds, an ointment base composed of pure stearin (if such substance were available) but softened by an admixture of liquid petrolatum would preserve the iodine satisfactorily. Cocoanut oil (iodine No. 8) ought to be suitable also if mixed with hard paraffin.

Since the literature was not sufficiently concordant to warrant positive conclusions concerning the stability of ointments containing free iodine, it seemed worth while to conduct experiments with preparations of known origin. Accordingly a number of preparations containing free iodine were made under varying conditions and each was assayed for its free iodine content immediately after its manufacture and from time to time later.

Leaf lard of the best quality obtainable was purchased from a butcher. This was rendered in an open dish on the steam bath. The preparation was of a fine color, and uniform consistence and had a faint but not unpleasant odor. Two specimens of lard were furnished by the research department of Armour and Company. An effort was made to procure specimens of lard having iodine absorption numbers as far apart as possible, *i. e.*, one with a low and the other with a high iodine value. This was done in order to determine whether the keeping qualities of the ointments prepared from the two would be alike.

One of the specimens (*a*) was described as

"Natura lard; iodine value, 57.1. Leaf lard used exclusively for butterine and benzoinated lard."

The other specimen was described as

"Prime steam lard. Good, commercial grade of lard for general use; iodine value, 69.0."

The iodine absorption numbers of the three specimens were determined by the U. S. P. process, and were found to be as follows:

Laboratory rendered specimen.....	57.1
Armour specimen ( <i>a</i> ).....	57.65
Armour specimen ( <i>b</i> ).....	67.55

Each specimen was benzoinated according to the process described in the U. S. P. IX and 100 gm. of iodine ointment

were prepared from each according to the U. S. P. process. Another specimen was made from benzoinated lard and iodine only<sup>11</sup> without the addition of either glycerin or potassium iodide. This was made to contain 4 per cent. of iodine.

Immediately after preparation each of these iodine ointments was assayed for free iodine, and each was reassayed at intervals later. The method for the determination of iodine in the ointment was that employed in this laboratory for the determination of iodine in Iocamfen Ointment.<sup>12</sup> It is essentially the same as was employed by Pullen for the determination of uncombined iodine in iodine ointment.<sup>13</sup> As carried out in this laboratory for iodine ointment it is as follows:

From 5 to 8 gm. of the ointment were weighed in a small porcelain capsule, the capsule and contents placed in a 16 oz. salt mouth bottle together with 20 c.c. of chloroform, 10 c.c. of potassium iodide solution and 40 c.c. of water. Tenth-normal sodium thiosulphate was slowly added with agitation until the pink color of the chloroform layer had nearly disappeared. A little soluble starch was then added and the titration continued until a blue color in the aqueous layer could no longer be obtained by repeated shaking.

The findings for the several assays are tabulated herewith:

TABLE 1.—IODINE CONTENT OF IODINE OINTMENTS

Age at time of assay	U. S. P. ointment from laboratory rendered lard (% I)	U. S. P. ointment from commer- cial lard Grade I (% I)	U. S. P. ointment from commer- cial lard Grade II (% I)	Ointment from lard and iodin only (lab- oratory rendered lard) (% I)
Freshly made .....	3.32	3.26	3.30	0.32
After 3 days.....	3.25	....	....	0.23
After 7 days.....	2.99	3.17	3.15	....
After 3 weeks.....	3.01	3.19	3.07	....
After 7 weeks.....	3.12*	3.10	3.02	....
After 3 months.....	2.98	2.88	2.88	....

\* This slight rise in iodine content followed by a fall could not be accounted for. The specimen was believed to have been very thoroughly mixed at the time of manufacture.

11. In order to facilitate the incorporation of the iodine with the fatty base the iodine was first powdered by trituration with alcohol and drying the powder in the air.

12. Rep. Chem. Lab., A. M. A., 1916, 9, 118.

13. Pharm. Jour., 1912, 89, 610.

That the fatty constituents of the ointment contained iodine after the preparation had been made for some time was demonstrated. Some of the material was examined as follows:

A portion of the ointment which had been made for nearly three months was shaken in a separator with chloroform and a dilute mixture of potassium iodide and sodium thiosulphate solutions. After all of the free iodine had been removed the chloroformic solution of the fats was washed several times with a very dilute solution of sodium thiosulphate. The chloroformic solution was filtered, evaporated and the residue dried over sulphuric acid.<sup>14</sup>

The separated fat was then tested for iodine by Kendall's method.<sup>15</sup> It was found to contain iodine in considerable amounts, but quantitative determinations were not made.

The Pharmacopeia of the Netherlands directs that iodine ointment shall contain 3 per cent. of potassium iodide and 2 per cent of iodine instead of equal proportions (4 per cent. of each) as prescribed by the U. S. Pharmacopeia. Likewise the French Pharmacopeia directs that 10 per cent. of potassium iodide and only 2 per cent. of iodine shall be used. Both of these pharmacopeias use water instead of glycerin as the solvent. Loose combinations of iodine and potassium iodide, such as is represented by the compound having the formula  $KI_2$ , have been described. The quantity of potassium iodide prescribed by the U. S. Pharmacopeia for the preparation of iodine ointment is not sufficient to form such a com-

14. The resultant fatty residue was of a brownish-green color. It no longer had either the taste, color or odor of lard. It was noted that the fats, after removal by this method from the freshly prepared ointment, were nearly white. As the ointment aged the fat became successively darker in color.

15. The method depends upon the conversion of all of the iodine compounds into iodate by fusion with sodium hydroxide and oxidation with potassium nitrate. The melt is dissolved in water, a little sodium bisulphite added, the solution cooled and neutralized with phosphoric acid, using methyl orange as indicator. An excess of bromine water is added, and the mixture boiled to expel carbon dioxide and bromine. A little sodium salicylate is added, the solution cooled, an excess of potassium iodide added, and the liberated iodine titrated with tenth-normal sodium thiosulphate in the usual way. One sixth of the iodine found is obtained from the material assayed, the balance being furnished by the potassium iodide added.—*Jour. Biochem.*, 1914, 19, 251.

pound as  $KI_2$  with all of the iodine directed to be used. Since some of the pharmacopeias use larger proportions of potassium iodide (more than sufficient to form the compound,  $KI_2$ ), it seemed worth while to determine whether an ointment containing a greater proportion of potassium iodide than that required by the U. S. Pharmacopeia would be more stable than the official article. Accordingly a specimen was prepared to contain 4 per cent. of iodine, 8 per cent. of potassium iodide (twice the U. S. P. requirement), 12 per cent. of glycerin and 76 per cent. of lard. This was assayed for its free iodine content immediately after preparation, and found to contain 3.68 per cent. Nine days later it contained 3.70 per cent. Another specimen of the same iodine strength prepared from grade No. 2 of commercial lard assayed 3.69 per cent. at the initial assay, and seven days later 3.40 per cent. From these experiments it seems likely that the free iodine content of the U. S. Pharmacopeia iodine ointment could be raised somewhat by increasing the proportion of potassium iodide.

The results of these studies confirm the findings of Pullen and of Fried in all essential particulars. It appears that during the process of manufacture of iodine ointment about 20 per cent. of the free iodine goes into combination with the fatty constituents of the ointment. On standing for a month approximately an additional 5 per cent. goes into combination, after which there is practically no loss in free iodine content. In other words iodine ointment which is a month old is a relatively stable preparation. It appears to make no noticeable difference upon the rate and amount of iodine absorption whether the lard from which the ointment is made has a high or a low iodine absorption value. The composition of iodine ointment, which has been made sufficiently long to have reached equilibrium, is approximately as follows:

Free iodine .....	3 per cent.
Iodine combined with fat.....	1 per cent.
Potassium iodide .....	4 per cent.
Benzoinated lard (containing iodine)....	80 per cent.

The U. S. Pharmacopeia requirement that iodine ointment shall be freshly prepared when wanted appears to be unnecessary. Probably most pharmaceutical manufacturers are aware of this, for many of them include the preparation in

their trade lists. The presence of an iodide appears to be necessary, to prevent practically all of the iodine from entering into combination with the fat.<sup>16</sup>

16. In order to determine whether the iodine which is in combination with fat is absorbed through the skin, a few experiments were carried out. The dark-colored iodine-containing fat (obtained from the ointment and washed free from potassium iodide by the method described above) was rubbed thoroughly into the skin of the forearm. It was allowed to remain for four hours, after which the limb was scoured with soap suds. Beginning at the time of the application the urine was collected for forty-eight hours. This was evaporated to small bulk and the residue tested for iodine by Kendall's method. Small amounts of iodine were found. These findings were taken to indicate that the iodine-containing fat is absorbed to some extent by the skin. It is generally believed that potassium iodide is not absorbed by the unbroken skin. Therefore it seems reasonable to suppose that the principal iodine effects obtainable from iodine ointment are those due to the free iodine contained in the preparation, supplemented to a slight extent by the iodine which is contained in the fatty ointment base.—*Jour. Biochem.*, 1914, 19, 251.



## PART II

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### REPORTS ABSTRACTED AND REPRINTED FROM THE JOURNAL A. M. A. AND FROM THE REPORTS OF THE COUNCIL ON PHARMACY AND CHEMISTRY

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#### THE J. B. L. CASCADE TREATMENT

*Abstracted, with additions, from The Journal A. M. A., Jan. 6, 1917,  
p. 50)*

The J. B. L. Cascade Treatment is exploited by Charles A. Tyrrell, of New York City. The "treatment" consists of the self administration of rectal enemas by means of a rectal syringe called the J. B. L. ("Joy-Beauty-Life") Cascade. The syringe is sold by Tyrrell. The enemas contain a medicinal substance also sold by Tyrrell called the "J. B. L. Antiseptic Tonic." Concerning the "Antiseptic Tonic" Tyrrell says:

"This preparation is one of the most important features of the treatment, for it attacks and destroys the microbes of disease in their principal breeding place, and I cannot too strongly urge the importance of its use, for the destruction of these germs prevents disease and hastens a cure."

A woman who had received some of the advertising material sent out by Tyrrell wrote asking why the formula of the "Antiseptic Tonic" was not given. Here is what she was told:

"Note your comments as to the Antiseptic Tonic and your inquiry as to why we do not give the formula. We cannot see any good reason why we should do anything of this kind, and give it to the public in order that they may searc[e] the tonic from the druggist without any profit to ourselves for all time."

A specimen of J. B. L. Antiseptic Tonic was examined and the following report published in *The Journal*:

J. B. L. Antiseptic Tonic is a dirty white, faintly perfumed powder containing small blue particles. A 4-ounce package

retails for 50 cents. Dissolved in water it produces a faintly greenish-blue, turbid solution, having an alkaline reaction to litmus. Qualitative tests indicated the presence of a chlorid, a borate, sodium, small quantities of calcium and of a sulphate and traces of a nitrate. Alkaloids, ammonium salts, free boric acid and salts of copper and zinc were absent.

Quantitative determinations indicated that the composition of the specimen examined is essentially as follows:

Sodium chlorid (common salt).....	69.9 per cent.
Borax .....	29.6 per cent.
Calcium sulphate, hydrous (gypsum)...	0.5 per cent.
Color and perfume.....	traces

It seems probable that the gypsum found is not an intentionally added constituent, but that it occurs as an impurity or an adulterant in a cheap grade of borax used in preparing the mixture. The insolubility of the gypsum is responsible for the turbidity of the solution.

A preparation having all of the "antiseptic" and "tonic" properties of J. B. L. Antiseptic Tonic can be made by mixing 2.8 ounces of salt with 1.2 ounce of ordinary borax at a cost of not to exceed two-thirds of 1 cent.

Concerning the composition of the preparation *The Journal* commented in part as follows:

Here, then, we have  $2\frac{1}{2}$  ounces of table salt mixed with  $1\frac{1}{2}$  ounce of borax and sold for 50 cents! No wonder Tyrrell "cannot see any good reason" for giving the public the "formula" for his "Antiseptic Tonic" which "attacks and destroys the microbes of disease in their principal breeding place!"

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### BROM-I-PHOS

(Abstracted, with additions, from *The Journal A. M. A., Jan. 1, 1917, p. 2601*)

The laboratory took up the examination of Brom-I-Phos made by the National Drug Company, Philadelphia, because the Council on Pharmacy and Chemistry was investigating this preparation, and requested a report from the laboratory in regard to its composition.

Brom-I-Phos was submitted to the Council with a label bearing the following statement:

"ALCOHOL 25 PER CENT."	
COMPOSITION—Per Fluidounce.	
Iodin .....	1 gr.
Bromin .....	1 gr.
Phosphorus .....	8-100 gr.
Aromatic Base .....	q. s."

A request for further information in regard to the composition of Brom-I-Phos was sent to the National Drug Company. It was suggested that since the preparation cannot contain the stated amounts of free bromin, free iodin and free phosphorus, the form of combination in which these elements are present should be set forth. In reply, the firm said, first, that "Brom-I-Phos consists of Bromin, Iodin, Phosphorus, Glycerin, Wine, Water and Volatile Oils. The Iodin is rubbed up with a small percentage of Potassium Iodid and 95 per cent. Alcohol, which solution is mixed with a solution of Bromin and Spirits of Phosphorus which are combined with the base and aromatics." The manufacturer also admitted that phosphorus reacts with bromin and iodin and that other reactions might occur, but maintained that it was "justified in assuming the greater part, if not all of these elements, are actually existent in the nascent state," and asserted that its "printed formula complies with our working formula in point of quantities involved as well as existence of elements in an uncombined state."

Since the firm's reply was unsatisfactory in that it claimed that the greater part of these elements actually exists in Brom-I-Phos in the "nascent state," the preparation was examined to determine, if possible, the form in which the bromin and iodin are present. The presence of free phosphorus, free bromin and free iodin could not be demonstrated in Brom-I-Phos. Neither iodate nor bromate were present. A bromid and an iodid were present in large amounts. After dilution with water the addition of silver nitrate to an acidulated portion of the material gave an amount of silver halid, roughly agreeing with that which would be obtained had the claimed amount of bromin and iodin (together with some potassium iodid) been used in the preparation of Brom-I-Phos and in the process of manufacture become converted to bromid and iodid.

In view of the laboratory findings the Council held that the statement of composition was unsatisfactory and misleading in that it suggested that the preparation contained bromin, iodin and phosphorus in the free (elementary) state, and that the presence of the potent elementary phosphorus was especially suggested by the small amounts of "phosphorus" declared.

In declaring Brom-I-Phos not admissible to New and Nonofficial Remedies because of the misleading statements made in regard to its composition; because of the exaggerated therapeutic claims; and because the combination of bromin, iodin and phosphorus, or bromid, iodid and phosphate is irrational, the Council pointed out that the name does not indicate that Brom-I-Phos is an alcoholic preparation with iodid as its essential constituent, but suggests that phosphorus is an important constituent, whereas the amount of phosphate or phosphite, produced by the action of iodin on elementary phosphorus (if the amount of phosphorus used in making the preparation is correctly stated) is insignificant.

#### Details of Analysis

*Halogens*.—After evaporating the alcohol, diluting the solution with water and adding silver nitrate a pale yellow precipitate was given. This was collected in a weighed Gooch crucible, dried and weighed. The silver halid found was equivalent to 3.79 grains of iodin per fluidounce.

*Bromates and Iodates*.—These salts were proved to be absent by acidulating with sulphuric acid, adding potassium iodid and shaking the mixture with chloroform. A violet color was not produced. Upon addition of either potassium bromate or potassium iodate to the acidified solution and shaking with chloroform a violet color was given.

*Bromid*.—The dealecholized preparation was diluted with water, the solution acidulated with nitric acid and silver nitrate solution added. The precipitated silver halid was washed with hot water and dissolved so far as possible in warm, 10 per cent, ammonia water. The mixture was filtered and nitric acid added to the cold filtrate. An abundant, yellow precipitate was given. This was considered to be evidence of the presence of bromid.

*Iodid.*—The insoluble, pale yellow precipitate remaining, on treatment of the total silver halid with warm ammonia water, was considered to be evidence of the presence of an iodid. Some of the dealecoholized preparation was acidulated treated with ferric chlorid and the mixture shaken with chloroform. A violet color in the chloroform layer was obtained.

*Phosphorus.*—Free phosphorus was concluded to be absent after consideration of the following facts: (1) It is well known that phosphorus does not dissolve and cannot be held in solution in any appreciable quantity by 25 per cent. alcohol. Even when dissolved in boiling 95 per cent. alcohol considerable of the free phosphorus separates on cooling. (2) Further, spirit of phosphorus (which is made with absolute alcohol) is not stable if kept for any length of time in partially filled bottles. (3) Furthermore, on shaking some of the Brom-I-Phos with copper acetate solution no black color or black precipitate was produced. These facts, taken together, are considered sufficient proof that free phosphorus is not present.

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### LOW'S WORM SYRUP

(Abstracted, with additions, from *The Journal A. M. A.*,  
July 21, 1917, p. 225)

Specimens of Low's Worm Syrup made by Smith, Kline and French Company, Philadelphia, were examined. The preparation tested was a thick, dark brownish syrup having an odor and taste resembling Dr. Hand's Worm Elixir of the Hand Medicine Co., also sold by Smith, Kline and French Company. The presence of 11 per cent. of alcohol was declared.

"The advantage of Low's Worm Syrup over many other preparations is, that it is a *vegetable product*, and is not only most certain in its effects, but also most pleasant, being free from Castor Oil or other nauseous drugs. The alcohol present is essential as a solvent and preservative."

"It kills the worms and at the same time, by its cathartic principle, cleans them out of the system without pain, thereby avoiding the necessity of administering Castor Oil, or other unpleasant cathartics."

Further than this, the trade package furnished no information concerning the composition of the preparation. A considerable quantity of suspended matter was noted. This

was of a yellowish color, and, together with the therapeutic recommendations of the preparation, suggested the probable presence of santonin. Qualitative tests indicated the presence of santonin, alcohol, sucrose and extractives from some emodin-bearing drug, probably senna. The lack of a noticeable bitter taste indicated the absence in more than traces of many of the common laxatives, such as aloes or colocynth. If present, their quantities must be small. The purgative salts, such as Epsom salt, Rochelle salt, etc., were absent. Pomegranate and pink root, or their extractives, were absent. The quantitative examination was limited to the determination of santonin. The quantity found amounted to about 0.93 gm. per 100 c.c. of the preparation, or about 4.2 grains per fluidounce. Each drachm (teaspoonful) of the preparation therefore contains a little more than one-half grain of santonin.

In publishing the results of the above examination, *The Journal* commented in part:

"Santonin is a poison. One grain has produced serious poisoning in children and two doses of 1 grain each have been fatal. Total blindness and death are among the results that have followed the use of the drug. Headache, dizziness and convulsions, with stupor, loss of consciousness and death represent the train of symptoms following poisonous doses of santonin. As the chemists have shown, each teaspoonful of Low's Worm Syrup contains over half a grain of santonin. . . . The directions for children from three years of age and older calls for from 2 to 2½ teaspoonsfuls presumably four times a day, at least the wording of the label is so ambiguous that the mother may readily assume that that is what is meant. This means giving children from 4 to 6 grains of santonin daily!"

"The action of santonin, because of its excretion through the kidneys, is likely to be cumulative. Sollmann, in the 'Manual of Pharmacology,' warns that the drug should only be given under the supervision of a physician and states that half a grain may be given for two or three doses, according to age, to children of from two to five years of age 'and then not repeated for at least three days.' In spite of these facts, the Low's Worm Syrup circular urges mothers to lose no time, but

resort at once to Low's Worm Syrup' whenever their children are troubled with such symptoms as:

"Pain in the joints, 'all gone' feeling at the stomach, drowsiness, bad breath, picking at the nose, grinding of the teeth, a gnawing sensation of hunger, flashes of heat, chills or shivering, vertigo, disturbed sleep, startling dreams, want of appetite or excessive appetite, pain in the stomach or bowels, nausea, indigestion, costiveness and convulsions."

"The problem presented by such preparations as Low's Worm Syrup is simple in principle but, because of the power of entrenched wealth, difficult of application: There is no excuse in economics or morality for putting on the market as a home remedy preparations containing so dangerous a drug as santonin. So long, however, as the laws do not prohibit this the least a manufacturer with any decent regard for public health and safety can do is specifically to warn the purchasing public that the preparation contains a dangerous drug."

#### Details of Analysis

*Santonin.*—The method used for the determination of santonin in Low's Worm Syrup was essentially the same as that employed in the analysis of Dr. Hand's Worm Elixir. Gum-like substances were present in much smaller quantities than in the Worm Elixir. Consequently, on treating the mixture with warm alcohol and allowing it to stand, principally sucrose, rather than much gum separated. An attempt was made to shake out the santonin directly, with chloroform, but this gave lower results than by first precipitating with alcohol and the method was abandoned. The remainder of the analysis was carried out as described under Dr. Hand's Worm Elixir. From the contents of one bottle, 48.1277 gm. of material (equivalent to 38.5962 c.c.), 0.3765 gm. of santoninic acid was obtained, equivalent to 0.909 gm. of santonin per 100 c.c. of material. The contents of another bottle, 45.4881 gm. (36.2091 c.c.), gave 0.3666 gm. of santoninic acid, equivalent to 0.9433 gm. of santonin per 100 c.c. Average, 0.926 gm. of santonin per 100 c.c., or about 4.2 grains per fluidounce. By the direct shake-out method, a third portion of 44.7892 gm. of material (35.9204 c.c.) gave 0.2914 gm. of santoninic acid, equivalent to 0.2715 gm. of santonin, or about 0.774 gm. of santonin per 100 c.c. This result was discarded.

*Senna, Purgative Salts, Other Cathartics, etc.*—The tests for laxative substances, alkaloids, etc., were carried out essentially as described elsewhere under Dr. Hand's Worm Elixir.

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## DR. PIERCE'S ANURIC TABLETS

### A Fake Cure for Kidney Disease

(Abstracted from *The Journal A. M. A.*, Sept. 15, 1917, p. 30)

The Propaganda Department of THE JOURNAL asked the Chemical Laboratory to examine Dr. Pierce's Anuric Tablets, manufactured by the World's Dispensary Medical Association of Buffalo, N. Y. In due time the chemist submitted the following report:

#### CHEMIST'S REPORT

One-half dozen original bottles of "Dr. Pierce's Anuric Tablets" were submitted to the Chemical Laboratory for examination. Each bottle contained fifty red, kidney shaped coated tablets, weighing on an average 0.5 gm. ( $7\frac{1}{2}$  grains). Qualitative tests indicated the presence of acetate, carbonate, chlorid, iodid, phosphate (trace), salicylate, ammonia, calcium, iron (trace), potassium, sodium, emodin, aloin, quinin, hexamethylenamin and sugar. The amount of reducing sugar, calculated as dextrose, was about 35 per cent. Besides the substances mentioned above, there are evidences of other drugs being present. What some of these drugs are may be judged by tracing the so-called quotations appearing in the circular around the bottle. Although in many instances garbled, the statements evidently refer to such plant drugs as *Apocynum cannabinum* (Canadian hemp), *Eupatorium purpureum* (queen of the meadow).

Such a mixture as the foregoing is so irrational and foolish that a more exhaustive examination appeared a waste of time. But from the qualitative data alone, it can be seen that Anuric Tablets contain essentially sugar, an acetate, iodid and salicylate of either sodium or potassium, quinin, aloin and hexamethylenamin. Calcium carbonate is present as part of the coating.

#### THE JOURNAL'S COMMENT

Having received this report, THE JOURNAL discussed the false claims of Pierce's Anuric Tablets, and especially

exposed the fallacious uric acid and lithia arguments. Those who purchase a box of these tablets get, in addition to the fifty kidney-shaped red pills, a booklet that may be counted on to convince the average person that he or she has kidney disease. To demonstrate better the liberties that have been taken in compiling the advertising "literature" on Anuric Tablets, part of the matter that has been printed in the Anuric booklet, alleged to be a quotation from Lloyd and Felter, King's American Dispensatory, was compared in parallel with the original:

ALLEGED QUOTATION AS IT APPEARS IN THE ORIGINAL	APPEARS IN THE ORIGINAL
PEARS IN THE ANURIC BOOKLET	"Rheumatism yields to it when edema of a part of or whole of the body is present, or even where there is slight puffiness or glistening of the parts. Frequently it must be given with other antirheumatics.
<p>"Rheumatism yields to it, when a swelling of a part or whole of the body is present, or even where there is slight puffiness or glistening of the parts. It is a decided heart stimulant and has relieved the heart oppression due to smoking. It is also a decided antineuralgic, relieving sciatic, pleural and lumbar neuralgia. The most valuable remedy to relieve renal congestion or inflammation. It is one of the best remedies for acute inflammation of the upper passages of the nose and throat."</p>	<p>"Apocynum is a decided heart tonic. The conditions above named, and a dilated condition of the cardiac ventricles, point to its use. It is not the remedy where the circulation is excited, with hard, quick pulse. Dr. E. R. Freeman reports an inveterate case of angina pectoris benefited by it. Edema was a feature of the case. Dr. Waterhouse relieved the precordial oppression of a smoker with it. Dr. J. C. Kilgour declares it a decided antineuralgic, relieving sciatic, crural, and lumbar neuralgias. Prof. G. C. Gere asserts that it is the most valuable of deobstructors to relieve renal congestion in the second stage of tubular nephritis. Too much, however, must not be expected of it where there are structural changes of the vital organs. Acute inflammation of the upper laryngeal and post-nasal is specifically met by this drug, according to Prof. Webster being nearly as positive as phytolacca, and preferable when the irritation does not extend beyond those parts, and is readily brought on by slight exposure."</p>

By comparing the alleged quotation with the original, it will be seen that the Anuric concern, by separating phrases from their context, omitting qualifying clauses, featuring the

alleged virtues and ignoring the limitations of the drugs discussed, attempts to make out a case for what is said to be one of the ingredients of its "kidney cure."

Summed up, THE JOURNAL stated, it may be said that the selling scheme of Anuric Tablets is first to frighten those who have any vague aches and pains into the belief that something is wrong with their kidneys for which Anuric purports to be the one great remedy. The popular idea that urinary sediments and pain in the lower part of the back mean kidney disease, while false, is assiduously cultivated. It is unnecessary to tell physicians of the dangers of self-diagnosis and self-treatment in such a serious condition as true disease of the kidney. The public is wedded to the idea that every disease can be cured by taking something out of a bottle three or four times a day. The average man does not realize that in the treatment of kidney disease the hygienic and dietetic measures may be of vastly greater importance than drug therapy. Such measures mean a certain amount of discomfort to the patient, the breaking up of old habits, a readjustment of one's method of living. If a cure can be brought about by taking medicine, why should one subject oneself to the minor discomforts of a modified diet and a more or less exacting hygiene? Therein lies the danger and wickedness of all alleged cures of kidney disease.

Not only are "Anuric Tablets" foisted on the public under false and misleading claims, but, as alleged cures for diseases that should never be self-treated, they are fundamentally and essentially vicious.

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## ZIRATOL

(*Abstracted, with additions, from The Journal A. M. A., Oct. 6, 1917, p. 1191*)

Ziratol is sold by the Bristol-Myers Company of New York. According to the manufacturer:

"Ziratol is prepared from Phenols of the Naphthalene series and consists of a solution of such Phenols in a mixture of soap, water and glycerin."

The label on a purchased trade package stated that the preparation contains 32 per cent. of water and 30 per cent. of glycerin (both by weight) as inert constituents.

The specimen of Ziratol examined was a dark brown solution having a phenol-like odor. By extracting the product with ether and evaporating the solvent a brownish, crystalline residue was obtained, which responded to most of the tests for alpha-naphthol. The melting point of the residue was not correct for pure alpha-naphthol but the discrepancies probably may be accounted for by the presence of small quantities of impurities. The impure alpha-naphthol was present in considerable amounts. No other antiseptic was found. The presence of glycerol and soap was confirmed.

The quantitative examination indicated that the specimen of Ziratol examined has essentially the following composition by weight:

Alpha-naphthol .....	18 per cent.
Soap .....	20 per cent.
Glycerol and water, sufficient to make 100 per cent.	

The examination shows that Ziratol is similar in composition to Benetol which was found to be a glycerin-water-soap solution containing about 18 per cent. of alpha-naphthol (Rep. Chem. Lab., A. M. A., 1911, **4**, 82).

The above findings were reported to the Council on Pharmacy and Chemistry by a referee along with a discussion of the other claims made for the product. The Council held Ziratol inadmissible to New and Nonofficial Remedies because its composition is secret; because the phenol coefficient, determined according to the method of the Hygienic Laboratory, U. S. P. H. S., is not stated on the label; because the label and the circular accompanying the trade package advises its use by the public as a "vaginal douche," and because the claim that Ziratol is the "universal disinfectant" is exaggerated and unwarranted.

#### Details of Analysis

*Ether Extract (Alpha-Naphthol).*—A weighed portion of the material was diluted with water and the solution shaken with ether which had previously been washed with water. The ether was allowed to evaporate without heat in a place protected from dust and the residue dried over sulphuric acid. Constant weight could not be obtained as the residue continued to lose weight indefinitely. The weight of the residue arbitrarily taken as the ether extract was that at the beginning of which the loss for each twenty-four hours became

approximately constant. The ether extract was brownish in color and was evidently impure. On heating the residue with proper precautions a sublimate of white crystals was obtained. From 10.4706 gm. of material 1.9268 gm. of ether extract was obtained, equivalent to 18.42 per cent. The brownish ether-extract melted at 74 to 76 C. The sublimate obtained from this melted at the same temperature. Some of the material was recrystallized from boiling petroleum ether. The purified product melted at about 92 C. A market specimen of alpha-naphthol bearing the Merck label melted at 94 C. and the sublimate from this melted at the same temperature. A mixture of equal weights of alpha-naphthol and beta-naphthol (market material) melted at 77 to 82 C. With the exceptions of the melting point, the brownish ether-extract responded satisfactorily to the chemical tests for alpha-naphthol as given in the literature. The substance did not give any of the tests recorded for beta-naphthol. The tests used are given here-with:

To 1 c.c. of a 1 per cent. sucrose solution 2 drops of a 20 per cent. alcoholic solution of the ether residue were added and, after shaking, followed by 2 c.c. of sulphuric acid. A deep violet color resulted. On diluting the mixture with water a violet precipitate was produced.

With ferric chlorid solution the aqueous solution of the ether-residue gave a white precipitate which soon became pale violet.

To a few cubic centimeters of the aqueous solution a few drops of chlorin water were added. A white precipitate was formed which, after the addition of ammonia water, partially dissolved to form a turbid, greenish solution (distinction from beta-naphthol which under the same treatment ultimately gives a brown color).

*Soap.*—The solution from which the alpha-naphthol had been removed was partially evaporated, acidified with sulphuric acid and shaken with several successive portions of ether. The solvent was removed, allowed to evaporate spontaneously, the residue moistened with a few drops of absolute alcohol, the mixture warmed on the water bath and the residue afterward dried in an air oven at 60 C. From 10.4706 gm. of original material a residue of fatty acids weighing 1.9361 gm. was obtained, equivalent to 18.39 per cent. of fatty

acids. The balance of the work on the soap determinations is given in the succeeding paragraphs.

*Sodium and Potassium.*—The acid solution from which the fatty acids had been removed by ether was evaporated to dryness and the residue heated to low redness with a few drops of sulphuric acid. The residue was cooled, a little ammonium carbonate added and the mixture heated to constant weight. The total sulphate in the residue was then determined by weighing as barium sulphate and the proportions of sodium and potassium determined by calculation. From 10.4706 gm. of the material, 0.6038 gm. of the mixed sulphates of potassium and sodium was obtained, and this mixture gave 0.9528 gm. of barium sulphate, equivalent to 0.4026 gm. of sulphuric acid.

Since 1 gm. of anhydrous sodium sulphate is equivalent to 0.6899 gm. of absolute sulphuric acid and 1 gm. of potassium sulphate is equivalent to 0.5625 gm. of absolute sulphuric acid, the respective weights of anhydrous sodium sulphate and anhydrous potassium sulphate in a mixture of the two may be determined by solving the following equation:

$$0.6899x + 0.5625(w - x) = c$$

In which

$w$  = the weight of the mixed alkali sulphates found.

$x$  = the weight of the anhydrous sodium sulphate in the mixture.

$(w - x)$  = the weight of an anhydrous potassium sulphate in the mixture.

$c$  = the weight of the sulphuric acid found.

Substituting the values obtained in the analysis in the preceding formula the equation becomes:  $0.6899x + 0.5625(0.6038 - x) = 0.4026$  which gives  $x$  a value of 0.4941 gm. of anhydrous sodium sulphate — 0.1502 gm. of sodium (Na), or 1.53 per cent.  $(w - x) = 0.1097$  gm. of anhydrous potassium sulphate — 0.0492 gm. of potassium (K), or 0.47 per cent.

The total soap is the sum of the fatty acids (18.39 per cent.), the sodium (1.53 per cent.) and the potassium (0.47 per cent.), or 20.39 per cent.

*Glycerol.*—The solution from which the fatty acids had been removed was evaporated to a thick syrup, the residue treated with 25 c.c. of alcohol, the mixture allowed to stand over night, filtered and the filtrate evaporated to a thick syrup. A few drops of the residue were treated by the potassium bisulphite distillation method and the distillate tested for aldehydes by means of the decolorized fuchsin reagent. The reaction was positive. No quantitative determinations of the glycerol were made.

"HAINES' GOLDEN TREATMENT"

A Cruel Humbug Exploited as a Cure for the Liquor Habit

(Abstracted, with additions, from *The Journal A. M. A.*,  
Oct. 27, 1917, p. 1460)

"Dr. Haines' Golden Treatment" is sold and advertised by the Golden Specific Company of Cincinnati, Ohio. Before lying on the trade package carried with it a risk of prosecution, the "Golden Treatment" was sold as "Golden Specific." The preparation is one of the numerous fakes exploited as cures for the liquor habit which can be given secretly, curing the alcoholic in spite of himself. Here are some of the claims that have been made:

"Golden Treatment is Odorless and Tasteless — Any Lady Can Give It Secretly at Home in Tea, Coffee, or Food."

"Golden Remedy, the Great Home Treatment For Drunkards."

"Let no woman despair. The sure quick permanent cure for drunkards has been found. It is Golden Remedy. It has no odor. It has no taste. Just a little is put in the drunkard's cup of coffee or tea or in his food. He will never notice it, he will be cured before he realizes it, and he will never know why he abandoned the taste for liquor."

"Golden Remedy has cured some of the most violent cases in a day's time."

". . . a craving for liquor relieved in thousands of cases without the drinker's knowledge, and against his will."

Any one with an elementary knowledge of the treatment of alcoholism knows how cruelly false such claims as these are. Although the worthlessness of this product is obvious it was believed that its composition would be of interest, and an analysis of the stuff was made.

ANALYSIS

Original packages of "Dr. Haines' Golden Treatment for the Liquor Habit" (price, \$3 each), prepared by the Golden Specific Co., Cincinnati, were examined. Each box contained forty powders, the average weight of each powder being 75 gm. (11.5 grains). The material had a light brown color, a celery-like odor and a sharp taste. Under the microscope a few starch grain resembling those of ipecac were discerned; wheat starch was present in relatively large amounts. Qualitative tests demonstrated the presence of capsicum, lactose, starch, a small amount of resin and a very small amount of alkaloid. The amount of alkaloid was so

small that positive tests could not be obtained for the ipecac alkaloids. Emodin-bearing drugs were not present. The quantitative determinations were ash 1.47 per cent.; moisture (loss at 130°) 4.29 per cent.; lactose 47.5 per cent.; alkaloids 0.0003 per cent.

From the analysis it appears that Dr. Haines' Golden Treatment is composed essentially of milk sugar, starch, capsicum and a minute amount of ipecac.

THE JOURNAL, in concluding the article, agreed with Mr. Samuel Hopkins Adams, who, in his series in "The Great American Fraud," said:

"The Sunday newspapers and small weeklies teem with advertisements of 'drink cures,' which are supposed to exorcise the alcoholic craving when secretly given in tea or coffee. Few of these concoctions can be described as immediately dangerous, though none of them is really safe. All are swindles. They do not cure the drink habit."

#### Details of Analysis

*Alkaloids.*—Fifteen grams of the specimen, which was in the form of a fine powder, was placed in a 250 c.c. glass-stoppered Erlenmeyer flask, and shaken with 150 c.c. of ether. After a short time, 5 c.c. of ammonium hydroxid solution was added, and the flask shaken intermittently for two hours. Fifteen c.c. of distilled water was added, the contents were vigorously agitated, and then allowed to stand over night. One hundred c.c. of the ether solution (representing 10 gm. of the drug) was decanted through a peldorf of purified cotton into a separator, the procedure of shaking out being the same as described under *Belladonnæ Radix*, U. S. P. IX. The residue from the final ether extraction was dried at 60° C. and weighed 0.0034 gm., equivalent to 0.0003 per cent. The material gave a faint positive test for alkaloids with iodin test solution, potassium mercuric iodid and phosphotungstic acid. Tests for emetin and cephaeline applied to this residue were not conclusive.

*Lactose.*—4.7345 gm. of the specimen was treated with 100 c.c. of cold water. It was filtered through cotton, with vacuum, washed well, and then through filter paper, with vacuum, and with washings made up to 250 c.c. After standing for a number of days, the solution gave a rotation in a 200 mm. tube of 0.94 degrees. This is equivalent to 47.5 per cent. lactose.

*Moisture.*—3.2408 gm. of the specimen was dried in an electric oven at 130 C. for two hours. The loss in weight was 0.1393 gm., equivalent to 4.29 per cent.

*Ash.*—1.4055 gm. of the specimen was incinerated over a Meeker burner. The amount of ash was 0.0208 gm., equivalent to 1.47 per cent.

### VENOSAL

(Abstracted, with additions, from *The Journal A. M. A.*, Jan. 5, 1918,  
p. 42)

The Council on Pharmacy and Chemistry of the American Medical Association took up for consideration "Venosal," one of the products of the Intravenous Products Company. Its composition has been variously and obscurely described.

"Venosal is a sterile solution representing 1 gm. (15.4 grains) of salicylates in combination."

"Venosal is a sterile solution representing 1 gm. (15.4 gr.) of salicylates in combination, together with colchicum."

"This is a product for intravenous use. The composition of which is Sodium Salicylate, 15.4 grs. (1 gm.). Iron Salicylate a minute quantity and the equivalent of approximately 2 grs. Dried Colchicum Root."

None of these "formulas" gives the quantity of the product containing 1 gm. of salicylate, etc., but presumably it refers to the contents of 1 ampule, or 20 c.c. Hence a chemical examination was made.

In the original box were six 20 c.c. ampules, containing a purplish red solution. The label contained the following statement:

A sterile solution representing 1 gm. (15.4 grs.) salicylates and approximately 2 grs. (130 mg.) dried colchicum root. Dose, 20 c.c.

Quantitative estimations were made for salicylic acid, iron and alkaloids. As a result, Venosal may be said to contain in each 20 c.c. (ampule):

Sodium salicylate .....	0.999 gm.
Iron (Fe) .....	0.0018 gm.
Colchicum root (calculated from colchicin found) ..	0.125 gm.

The purple color is due to the small amount of iron in the presence of salicylate.

The Council, in its report, said:

Venosal is recommended for the treatment of "rheumatism," meaning, the context would indicate, infectious rheumatic fever. As colchicum has no special action on

this disease, and as there is no apparent reason for the employment of the trace of iron present, these additions in fixed proportions are unscientific, if not absurd.

The whole question of the justification of using salicylates intravenously is open to grave doubt. Since it is possible to obtain the salicylate effects promptly and certainly by oral administration, the inherent dangers of intravenous medication render its routine employment unwarranted. A further objection to Venosal, especially at this time when economy is a national policy, is the unnecessarily high expense of Venosal itself and of its administration.

The Council declared Venosal ineligible for inclusion in New and Nonofficial Remedies because of conflicts with Rule 1 (indefinite chemical composition), Rule 6 (therapeutic exaggerations), and Rule 10 (unscientific composition).

#### Details of Analysis

*Salicylic Acid.*—Ten c.c. of the sample were placed in a separatory funnel, a few drops of ammonia water added, and the solution shaken with three separate portions of chloroform. The aqueous solution was then acidified with hydrochloric acid, and chloroform added. The layer of chloroform was drawn off through a cotton pledge into a tared "half-beaker." The solution was extracted three times with chloroform. The chloroform was evaporated in a current of warm air. The residue was weighed, and then titrated with standard alkalie. (a) Ten c.c. yielded 0.429 gm. residue. This required 3.1 c.c. of normal sodium hydroxid solution, having a factor of 1.006, which is equivalent to 0.430 gm. salicylic acid. (b) Ten c.c. yielded 0.429 gm. residue. This required 3.1 c.c. of normal sodium hydroxid solution, equivalent to 0.430 gm. salicylic acid.

*Iron.*—Ten c.c. of Venosal (which contains 0.5 gm. sodium salicylate) was delivered into a colorimeter tube and diluted to 100 c.c. Into a second colorimeter tube were introduced 0.5 gm. of sodium salicylate and 96 c.c. of water. To the second tube was added very dilute ferric chlorid solution until the colors matched. The ferric chlorid solution was prepared by diluting 1 c.c. of a 10 per cent. ferric chlorid ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) solution to 200 c.c. Each c.c. of this solution

contained 0.0001 gm. iron. It required 4 c.c. to match 10 c.c. of Venosal. Calculating, each ampule of Venosal contains 0.0008 gm. iron.

*Alkaloids.*—Fifty c.c. of Venosal were placed in a separatory funnel, made alkaline with ammonia water, and shaken with three successive portions of chloroform. The chloroform was filtered through cotton into a tared "half-beaker," and then evaporated in a current of warm air. The residue weighed 0.0014 gm. Considering colchicum root to contain 0.45 per cent. colchicin, then 20 c.c. of Venosal would contain the equivalent of 0.125 gm. of colchicum root.

### SURGODINE

(Abstracted, with additions, from *The Journal A. M. A.*, Jan. 27, 1918, p. 257)

At the request of the Council on Pharmacy and Chemistry, the laboratory made an examination of Surgodine, a proprietary preparation marketed by Sharp and Dohme, Baltimore, Md.

The advertising pamphlet states that Surgodine is a solution of 2.25 per cent. of iodin in alcohol, containing no alkaline iodid but miscible with water in all proportions. Sharp and Dohme also assert that this preparation is superior for surgical use to the official tincture of iodin because of the absence from it of potassium iodid. All together, the claims made for Surgodine are suggestive that it is similar to Minson's Soluble Iodin (Rep. Com. Pharm. Chem., 1917, 152) and Burnham's Soluble Iodin (Rep. Chem. Lab., A. M. A., 1915, 8, 50).

An original bottle of Surgodine was purchased on the open market in November, 1917. The bottle contained a dark brown liquid resembling, in appearance, tincture of iodin, U. S. P. The specific gravity of the liquid was 0.8935 at 15.6 C. The preparation was miscible with water in all proportions. Qualitative tests indicated the presence of iodin, alcohol and acid. On evaporation of 5 c.c. of the product on a steam bath, practically no residue remained, showing absence of appreciable amounts of mineral salts.

Quantitative estimations yielded the following data:

Iodin (by saponification) .....	4.34 gm. per 100 c.c.
Iodin (by Kendall's method) .....	4.25 gm. per 100 c.c.
Iodin (free) .....	2.51 gm. per 100 c.c.
Iodid (calculated to hydrogen iodid) .....	1.32 gm. per 100 c.c.
Acidity (calculated to hydrogen iodid) .....	1.79 gm. per 100 c.c.
Alcohol (by volume) .....	91.8 per cent.

On the basis of the preceding findings, the laboratory reported to the Council:

Surgodine is an alcoholic liquid (containing 91.8 per cent. alcohol by volume) containing free iodin, combined iodin, and free acid, probably hydrogen iodid (hydriodic acid). Quantitative estimations gave 2.51 gm. free iodin per 100 c.c. and 1.79 gm. combined iodin (the greater part apparently was present as hydrogen iodid).

After consideration of the laboratory's report of the claims made for the preparation, the Council declared Surgodine inadmissible to New and Nonofficial Remedies, because its composition is secret; because the therapeutic claims made for it are exaggerated and unwarranted, and because it is an unessential modification of the official tincture of iodin.

#### Details of Analysis

*Iodin Total.*—1. Saponification Method: A sample of Surgodine was saponified with alcoholic potassium hydroxid solution according to the method described in the Reports of the Chemical Laboratory, A. M. A., 1916, **9**, 118, except that a reflux condenser was employed instead of the pressure flask. The iodid was determined as silver iodid, and calculated to iodin. (a) Five c.c. yielded 0.4021 gm. of silver iodid, equivalent to 4.34 gm. of iodin per 100 c.c. (b) Five c.c. yielded 0.4010 gm. of silver iodid, equivalent to 4.33 gm. of iodin per 100 c.c.

2. Kendall's Method: A sample (5 c.c.) of Surgodine was saponified, according to a slightly modified method of E. C. Kendall (Jour. Am. Chem. Soc., 1912, **34**, 894), with 30 c.c. of 20 per cent. sodium hydroxid solution in a pressure bottle for about an hour, transferred to a 500 c.c. Erlenmeyer flask, and acidified with phosphoric acid. The liberated iodin was titrated with tenth-normal sodium thiosulphate solution. The number of cubic centimeters of sodium thiosulphate solution consumed divided by 6 gave the equivalent of the amount of iodin present. (a) Five c.c. of Surgodine took 100.3 c.c. of tenth-normal sodium thiosulphate solution, equivalent to 4.24 gm. of iodin per 100 c.c.

*Iodin (free).*—Two 5 c.c. portions of Surgodine required 9.55 c.c. of tenth-normal sodium thiosulphate solution, equivalent to 0.1256 gm. of iodin, or 2.51 gm. per 100 c.c.

*Acidity.*—This was determined by two different methods: First, by titration, with alkali, of the colorless solution obtained after titration of the uncombined iodin, and, second, by shaking out the iodin from an aqueous solution of Surgodine with four portions of carbon disulphid and titrating the aqueous layer with standard alkali.

1. After titration of uncombined iodin in 5 c.c. of Surgodine, methyl orange was added and the liquid titrated with fiftieth-normal sodium hydroxid solution. In this determination, 34.35 c.c. of the standard alkali were required, equivalent to 1.74 gm. of hydrogen iodid per 100 c.c. In another determination, 5 c.c. of Surgodine required 35.7 c.c. of fiftieth-normal sodium hydroxid solution, equivalent to 1.81 gm. of hydrogen iodid per 100 c.c., an average of 1.77 gm. of hydrogen iodid per 100 c.c.

2. Five c.c. of Surgodine were added to 50 c.c. of water in a separatory funnel, and the free iodin shaken out with four 20 c.c. portions of carbon disulphid. The aqueous layer was titrated with fiftieth-normal sodium hydroxid solution. To neutralize the acidity, 36.1 c.c. of fiftieth-normal sodium hydroxid solution were required, equivalent to 0.0905 gm. of hydrogen iodid, or 1.80 gm. per 100 c.c.

*Hydrogen Iodid.*—In order to determine the extent of the acidity due to hydrogen iodid, the following experiment was performed: Five c.c. of Surgodine were mixed with 100 c.c. of water, and the mixture was shaken with several portions of chloroform until the aqueous layer was colorless, indicating the removal of the uncombined iodin. The aqueous layer was then distilled in the presence of sulphuric acid and ferric ammonium sulphate into potassium iodid solution, and the latter titrated with tenth-normal sodium thiosulphate solution. (a) It required 5.1 c.c. of tenth-normal thiosulphate solution, equivalent to 1.306 gm. of iodin per 100 c.c., or 1.319 gm. of hydrogen iodid. (b) Five c.c. of Surgodine required 5.25 c.c. of tenth-normal sodium thiosulphate solution, equivalent to 1.33 gm. of hydrogen iodid per 100 c.c. These results strongly suggest that the total acidity is not due to hydrogen iodid alone. After titration of the iodin in the foregoing, an odor suggestive of acetic acid was noticeable in the titration flask. However, no test for acetic acid could be obtained.

## PART III

### REPORTS NOT PREVIOUSLY PUBLISHED

#### COMMERCIAL PELLETIERIN TANNATE

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Inquiries were received concerning the composition of Pelletierin Tanret and the various products sold as "Pelletierine Tannate." Although a substance is described in the U. S. Pharmacopeia under the name of "Pelletierine Tannate," the literature concerning the product is extremely meager, and only one brief reference to any examination of the market products was found.

In 1913, Smith<sup>1</sup> reported that the several products at that time on the market under the name of pelletierin tannate differed more or less from the preparation described in the U. S. Pharmacopeia (VIII). They were usually incompletely soluble in water and in alcohol, and the prescribed color tests were not adequate, the reactions being obscured by the tannic acid or other organic matter. On extraction of the alkaloids by shaking with chloroform in alkaline solution, acidulating the chloroformic extracts with hydrochloric acid, evaporating, and drying the nonvolatile material remaining, residues of alkaloidal hydrochlorides ranging from 17 to 20 per cent. were obtained. These residues responded readily to the selenous acid test of the U. S. Pharmacopeia, but produced only slightly yellow colors with sulphuric acid.

The alkaloids of pomegranate bark, *Punica granatum*, are known in this country as punicin, isopunicin, pseudopunicin, methylpunicin and isomethylpunicin. In European literature the name "pelletierine" (with its combinations) is used in place of punicin. This came about because the discoverer of alkaloids in pomegranate bark, (Tanret) named the principal one "pelletierine" in honor of the French chemist, Pelletier, and the name is still retained for the most part by European writers.

Pomegranate bark and decoctions from it have been used as a teniafuge for many centuries. Because of the large proportion of tannin in the drug, and because of uncertain

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1. Smith: Jour. Am. Pharm. Assn., 1913, 2, 70.

strength, vomiting is sometimes induced. Consequently the salts of the alkaloids from the drug are preferred, those commonly used being the sulphate and the tannate. The pelletierin tannate described in the U. S. Pharmacopeia is said to be a mixture in variable proportions of the tannates of the four chief alkaloids of pomegranate. Pelletierin sulphate is described in the French Pharmacopeia (1908), and a method for its preparation is given. It is said to be chiefly composed of punicin (pelletierin) sulphate and isopunicin (isopelletierin) sulphate. The teniafuge properties of pomegranate are believed to lie chiefly in the punicin and isopunicin constituents; consequently the activity of the various crude commercial mixtures of the salts of the pomegranate alkaloids would depend on the relative proportions of these two alkaloids present.

In view of the inquiries received and of the very meager information in the literature, it was considered worth while to examine the market supply of pelletierin tannate. A specimen of each of the available market brands of pelletierin tannate (and also specimens of Pelletierin Tanret) were purchased. Specimens of pelletierin tannate also were obtained from some of the manufacturers, and these were included in the examination. The brands of pelletierin tannate examined were sold under the labels, respectively, of Merck and Company, the Mallinckrodt Chemical Works and the Powers-Weightman-Rosengarten Company. The material was in 5-grain and 15-grain packages. The contents of each package of the several brands were weighed, and the amount present in each case was found to agree with the quantity claimed.

The material from the several specimens was an amorphous, pale-yellow powder having a faint, conium-like odor. It was incompletely soluble in water or alcohol; practically completely soluble in very dilute hydrochloric acid; less soluble in more concentrated solutions of hydrochloric acid, and insoluble in chloroform or ether. The material insoluble in moderately dilute hydrochloric acid was of a resin-like consistence. The precipitate (in such of the specimens as gave it) was collected on a filter, washed with water, suspended in water, decomposed with sodium hydroxid, the mixture shaken with chloroform, the solvent evaporated in presence of hydrochloric acid, the residue taken up in water, and the solution tested for alkaloids by the usual reagents. The tests were negative, or at most showed only traces of alkaloids. The acid filtrate containing the soluble portions of the original material gave the usual tests for alkaloids.

On ignition, the material yielded only traces of ash. On being dried over sulphuric acid, the salt lost about 5 per cent. of moisture; when the desiccated substance was afterward exposed to moist air, it absorbed moisture. Solutions of the salt in very dilute hydrochloric acid were precipitated by the usual alkaloidal reagents. None of the specimens conformed to the U. S. Pharmacopeia requirement for foreign alkaloids. This test is as follows:

Platinic chloride T. S. produces no precipitate in a cold solution of about 0.1 Gm. of Pelletierine Tannate in a mixture of 4 mils of distilled water and 1 mil of diluted hydrochloric acid (*foreign alkaloids*). —

A noticeable precipitate was given when this test was carried out. The precipitate was collected, washed, decomposed by sodium hydroxid solution, the mixture shaken with chloroform, the solvent evaporated in presence of hydrochloric acid, the residue taken up in water, and the solution tested for alkaloids by the usual reagents. None were found. This indicates that the precipitate with platinic chlorid is not a foreign alkaloid. From this it seems probable that the U. S. P. test is fallacious. In other words, there appears to be some substance or substances present in some of the brands of commercial pelletierin tannate which are precipitated from dilute hydrochloric acid solution by platinic chlorid solution, yet which are not themselves alkaloidal in nature.

It seemed desirable to determine the proportion of alkaloids in each of the several specimens. Because of the volatile nature of some of the bases in pomegranate, it is not feasible to weigh the total alkaloids as such. Consequently, after being shaken out with chloroform, usually they are converted into their hydrochlorids by adding an excess of hydrochloric acid to the chloroform extract, evaporating, and weighing as alkaloidal hydrochlorids. As carried out in these determinations, the method is as follows:

From 0.5 to 1 gm. of the material was weighed into a separator, 15 mils of water added, followed by a slight excess of sodium hydroxid solution, and the mixture shaken with successive small portions of chloroform until all of the alkaloid had been removed. The ether extracts were united, washed with a little water, evaporated in presence of a slight excess of hydrochloric acid, and the residue dried at 60 C. and weighed.

RESULTS ON TESTS OF VARIOUS SPECIMENS

Brand	Solubility in Water	Loss Over $\text{H}_2\text{SO}_4$	Ash (Per Cent.)	Alkaloidal Chloroids (Per Cent.)	Miscellaneous	
					Very dilute HCl	PbCl <sub>4</sub> test
Merck, market	Incompletely soluble	5.61	0.015	0.031	Clear solution	Precipitate
Merck, submitted	Incompletely soluble	12.18	4.00	.....	Incomplete Resin like precipitate	Precipitate
M. C. W., market	Incompletely soluble	43.43	4.15	.....	.....	Precipitate
M. C. W., submitted	Incompletely soluble	78.82	4.70	0.02	17.38	Solution clear
F. W. R., market	Incompletely soluble	4.06	4.70	.....	19.18	Incomplete precipitate
Pelletierin Tincture	Soluble	Mostly insoluble (sucrose)	.....	0.0161	Sp. Gr. 1.2867 25 C.	Residue on drying at 100 C., 0.73 per cent.
			.....	.....	0.826	25 C.
			.....	.....	0.818	25 C.

The values found ranged from 17.3 to 22.7 per cent. The findings for the several specimens may be found in the accompanying table.

Since the alkaloids of pomegranate are supposed not to possess equal physiologic activity, an attempt was made to determine the proportions of the relatively more active ones by separating the total isolated alkaloids into groups. The method described in the literature is to add sodium bicarbonate to the salts of the mixed alkaloids and shake with chloroform. The solvent is drawn off and evaporated in presence of a little hydrochloric acid. Methylpunicin and pseudopunicin are said to be removed. Potassium hydroxid is added to the residue in the separator, and the mixture shaken again with chloroform. This treatment is said to remove punicin and isopunicin. An attempt to separate the mixed alkaloids from the commercial brands of pelletierin tannate by this method was tried after the hydrochlorids of the total alkaloids had been prepared and weighed. The results were unsatisfactory, and the method was abandoned.

#### PELLETIERIN TANRET

The exact composition of Pelletierin Tanret appears to be a secret, although it is generally supposed to contain tannates of some of the pomegranate alkaloids. No report of an analysis of the product was found in the literature, although a careful search was made.

Pelletierin Tanret is a thick, very pale yellow syrup having a faint, aromatic odor and a sweet, somewhat astringent, taste. Its specific gravity at  $\frac{25}{25}$  C. was found to be 1.2867.

The contents of the several individual packages varied widely in weight. Of six packages weighed, the maximum weight was 131.1 per cent. of the minimum. The diluted syrup had a neutral reaction toward litmus paper. Qualitative tests indicated the presence of a sulphate, sucrose, tannin and pomegranate alkaloids. Anthracene purgatives, such as aloes, cascara and rhubarb, and the heavy purgatives, such as Rochelle salt and Epsom salt, were absent. The alkaloids were determined by the following method:

Most of the sugar was precipitated from the syrup by 25 volumes of ether alcohol mixture (alcohol 90 per cent.,

ether 10 per cent.). The solvent was decanted from the precipitate, evaporated to a small volume, and the residue taken up in water. The solution of the residue in water was made alkaline with potassium hydroxid solution, shaken with ether-chloroform mixture, the solvent evaporated in the presence of hydrochloric acid, and the residue dried and weighed as alkaloidal hydrochlorids. The precipitated sugar was dissolved in 10 c.c. of water, and the sugar again precipitated by the ether-alcohol method. The alkaloidal extraction process described above was repeated, and the weight of alkaloidal hydr. chlorid found was added to the value obtained from the first extraction.

From the contents of one trade package of the preparation (26.3112 gm.), 0.2102 gm. of alkaloidal hydrochlorids was obtained by the first precipitation process and 0.0071 gm. by the second, or a total of 0.2173 gm. This is equivalent to 0.826 per cent. From 29.1960 gm. of material (the contents of another trade package), 0.2377 gm. of alkaloidal hydrochlorids was obtained from the first precipitation process, and 0.0012 gm. from the second, or a total of 0.2389 gm. This is equivalent to 0.818 per cent. The average is 0.822 per cent., calculated as alkaloidal hydrochlorids, or 0.653 per cent. as pelletierin alkaloid. From this it seems possible that the variability in the content of the several packages lies in the amounts of sugar contained therein rather than in the quantity of medicinal ingredients. Owing to lack of time, this phase of the subject was not further investigated.

Sulphate in the contents of one trade package of the preparation was determined in order to ascertain whether all of the alkaloid is present as sulphates or as a mixture of tannates and sulphates. From 9.6900 gm. of material, 0.0557 gm. of barium sulphate was obtained, equivalent to 0.059497 gm. of alkaloid calculated as pelletierin, or 0.614 per cent. This is approximately the value obtained by the shaking-out process. This, together with the presence of small amounts of tannin, point to the probability that pelletierin tanret consists principally of a crude mixture of the sulphates of the several alkaloids of pomegranate dissolved in syrup.

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"BYRON BARBER'S PNEUMONIA SPECIFIC"

A specimen, said to be "Byron Barber's Pneumonia Specific" (Byron Barber, Mineral Wells, Texas), was sent

in by a correspondent and was not contained in an original package.

The "Specific," according to the directions, is made into a decoction or "tea." It is directed to be administered for "Pneumonia, Pleurisy, severe Colds, pains in the chest, aching and soreness in limbs, or flesh, menstrual obstructions, confining patient to the house" in amounts "to keep the patient sweating moderately day and night until cured."

The "Pneumonia Specific" was in the form of a powder having a sharp spicy taste, suggestive of ginger or cinnamon. A microscopic examination of the specimen showed starch granules characteristic of ginger. Bast fibers and starch granules characteristic of cinnamon were also present. Oil globules were visible. This microscopic examination makes it probable that the preparation consists essentially of powdered cinnamon and powdered ginger.

A chemical examination indicated the absence of heavy metals and alkaloidal substances. The substance yielded a small amount of white ash, which apparently was not decomposed by acids; indicating the absence of organic salts, which yield carbonate on ignition.

From the preceding examination it is probable that the substance is essentially a mixture of powdered cinnamon and powdered ginger.

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#### STANDARDS FOR BETANAPHTHYL BENZOATE

With the request to revise the New and Nonofficial Remedies description of betanaphthol benzoate, if necessary, the referee of the Council on Pharmacy and Chemistry in charge of this product, submitted to the laboratory the following criticism of the description in New and Nonofficial Remedies, 1917:

In the test for the presence of betanaphthol, no mention is made of the quantity of benzonaphthol or of alkali to be employed. A test may or may not be obtained depending upon the quantities of materials involved. Is this not a test for benzoic acid as well as for betanaphthol? Is not the use of an alkali as strong as 10 per cent. likely to induce some hydrolysis of the benzonaphthol, resulting, of course, in a positive test for betanaphthol?

This criticism was sent to manufacturers of betanaphthyl benzoate whose products had been admitted to New and Nonofficial Remedies, with the request that they consider the criticism and aid in the establishment of suitable tests for identity and purity of betanaphthyl benzoate.

In reply the following suggestions were received:

One manufacturer stated that little or no hydrolysis of the betanaphthyl benzoate occurred if 1 gm. be shaken with 10 c.c. of a 10 per cent. solution of sodium hydroxid, providing the operation was carried out quickly and filtered immediately, and suggested a time limit of one minute for the operation.

Another manufacturer suggested that 1 gm. of betanaphthyl benzoate be shaken for one minute with 10 c.c. of an ice cold 5 per cent. solution of sodium hydroxid.

A third manufacturer stated that 5 and 10 per cent. solutions of sodium hydroxid were unnecessarily strong, while 1 per cent. solution of the alkali would be of sufficient strength to dissolve any betanaphthol present, and furthermore would insure no hydrolysis of the betanaphthyl benzoate.

An identity test was suggested by one firm, which consisted in saponifying with sodium hydroxid and the identification of the very characteristic betanaphthol and sodium benzoate.

Another firm suggested the color reaction of heating a chloroformic solution of betanaphthyl benzoate with alcoholic potash when a blue color will develop.

Two concerns advised testing for chlorids and sulphates as possible impurities in betanaphthyl benzoate.

The possibility of the presence of free benzoic acid led one firm to suggest that the neutrality of betanaphthyl benzoate ultimately be required.

In accordance with the above suggestions and after a careful study in the Laboratory, the monograph on betanaphthyl benzoate contained in New and Nonofficial Remedies, 1917, p. 197, has been revised. The tests for identity and impurities in the new monograph will read as follows:

Betanaphthyl benzoate occurs in colorless needles or as a white crystalline powder, tasteless and melting at 107 to 110 C.

Betanaphthyl benzoate is almost insoluble in water, but very soluble in alcohol and ether; also soluble in chloroform and fixed oils.

Betanaphthyl benzoate heated with a solution of potassium hydroxide in alcohol develops the odor of ethyl benzoate; on the addition of chloroform the mixture acquires a blue color.

Incinerate 0.5 gm. betanaphthyl benzoate; not more than 0.1 per cent. of ash remains.

Shake vigorously for one minute 1 gm. of betanaphthyl benzoate with 20 c.c. of a cold 5 per cent. aqueous sodium hydroxide solution and immediately filter. To 10 c.c. of the filtrate add 2 c.c. of chloroform and boil; no blue color is produced in the aqueous layer (uncombined betanaphthol). Carefully neutralize the remaining 10 c.c. of the alkaline filtrate, then add a few drops of ferric chlorid test solution previously diluted with two volumes of distilled water and neutralize, if necessary, with ammonia water; no pink precipitate is produced (uncombined benzoic acid).

Shake vigorously for one minute 0.5 gm. betanaphthyl benzoate with 5 c.c. of an aqueous 5 per cent. sodium hydroxid solution and filter. No blue color develops in the filtrate on the addition of a few drops of iodin test solution (alphanaphthol).

Shake vigorously for one minute 0.5 gm. betanaphthyl benzoate with 50 c.c. distilled water and filter. The filtrate should not be acid toward litmus. Five c.c. portions of the filtrate mixed with equal volumes of diluted nitric acid do not become turbid on the addition of 1 c.c. silver nitrate test solution (chlorid) or barium nitrate test solution (sulphate).

#### ELIXIR NOVO-HEXAMINE (UPSHER SMITH)

Elixir Novo-Hexamine is manufactured by Upsher Smith, of St. Paul, Minnesota. The preparation is a pale brown, syrupy liquid, having a sweetish, sour taste, an acid reaction and an odor resembling that of the so-called "sarsaparilla syrups." The label of Elixir Novo-Hexamine states that the preparation contains 2 per cent. of alcohol, but no sugar. According to the manufacturer, Elixir Novo-Hexamine is a

"stable, palatable, potent preparation of Novo-Hexamine, an acid compound of hexamethylenamine. Novo-Hexamine is far more active than hexamethylenamine, owing to its acid reaction."

Qualitative tests indicated the presence of sodium acid phosphate, hexamethylenamin and glycerol. Free formaldehyd

was shown to be present by the Jorissen test but its odor was not noticeable, possibly because of the presence of aromatics. Tests for alcohol were not made. The preparation was probably colored with caramel. Total nitrogen was determined, and the results calculated to hexamethylenamin. Phosphate and the acidity were determined, and the results calculated to sodium acid phosphate. An approximate estimation of the glycerol was also made. From the results of the examination it is concluded that the composition of Elixir Novo-Hexamine is approximately as follows:

Hexamethylenamin .....	12 gm.
Sodium acid phosphate ( $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ ) .....	11 gm.
Glycerol .....	23 gm.
Alcohol (manufacturer's claim) .....	2 per cent. by volume
Caramel coloring, flavor, etc., water, sufficient to make .....	100 c.c.

Since preparations containing hexamethylenamin and sodium acid phosphate in solution together might be expected to contain free formaldehyd, a measured quantity of Elixir Novo-Hexamine was diluted with water and the dilution colorimetrically compared with a known solution of formaldehyd after each had been treated by Jorissen's reagent, the colored solutions in each case being diluted to 50 c.c. with water. By this procedure the formaldehyd content was found to be equivalent to about 1 part in 416 parts of the preparation. Elixir Novo-Hexamine, therefore, appears to be merely a flavored and colored solution of sodium acid phosphate and hexamethylenamin in diluted glycerol.

The preceding report was submitted to the Council by a referee who reported that Elixir Novo-Hexamine was inadmissible to New and Nonofficial Remedies because of the following conflicts with the Council's Rules:

The manufacturer refuses to make any detailed statement as to the composition. The circular states that the elixir contains "Novo-Hexamine an acid compound of hexamethylenamin." Examination indicates that it is a mixture of hexamethylenamin and sodium acid phosphate. The preparation is therefore essentially a secret one. It is claimed that Elixir Novo-Hexamine permits the administration of hexamethylenamin in an acid vehicle without production of formaldehyd *in vitro*. But the chemical examination shows the presence of formaldehyd in the elixir.

The label bears the names of diseases in which the preparation is said to be indicated, thus tending to the ill-advised use of the preparation by the public.

The therapeutic misstatements are numerous. The preparation is said to be diuretic and "uratolytic." The best information indicates that hexamethylenamin has neither of these qualities to a notable degree. "Novo-Hexamine" is said to be more effective than hexamethylenamin because of its acid reaction. This is doubtless true of the elixir when it is added direct to urine. It is not necessarily true when it is given by mouth. In the latter case the efficiency depends whether or not enough of the acid phosphate is administered to render the urine acid. This cannot be assured by a routine dosage. The "indications" are altogether too optimistic; it cannot be said that hexamethylenamin is of "great value" in "whooping cough"; or that it "has succeeded" in "acute poliomyelitis," "epidemic polioencephalomyelitis" and "general paralysis."

The name does not sufficiently indicate the ingredients of this pharmaceutical mixture; but would tend to further the impression which the circular creates, namely, that we are dealing here with a definite new compound.

While it is often desirable to prescribe an acid phosphate during the use of hexamethylenamin it is not advisable to combine the two in one mixture; first, because this will tend to generate formaldehyd *in vitro*; and second, because the dosage of the two drugs generally needs to be varied independently of each other, so that fixed proportions are undesirable.

#### Details of Analysis

*Phosphoric Acid.*—Phosphoric acid was determined by heating a measured portion of the diluted solution with a mixture of nitric and sulphuric acids until all organic matter had been destroyed, precipitation of the phosphoric acid with ammonium molybdate, solution of the precipitate in ammonia water, adding magnesia mixture, collecting the ammonium magnesium phosphate and after heating, weighing the magnesium pyrophosphate in the usual way. Ten c.c. of the diluted solution, equivalent to 1 c.c. of the original material, gave 0.0916 gm. of magnesium pyrophosphate, equivalent to 9.876 gm. of anhydrous sodium acid phosphate ( $\text{NaH}_2\text{PO}_4$ ) per 100 c.c. A duplicate of 25 c.c., equivalent to 2.5 c.c. of undiluted material, gave 0.2209 gm. of magnesium pyrophos-

phate, equivalent to 9.526 gm. of anhydrous sodium acid phosphate per 100 c.c. Average: 9.701 gm. of anhydrous sodium acid phosphate per 100 c.c. This is equivalent to 11.16 gm. of hydrated sodium acid phosphate ( $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ ) per 100 c.c.

*Sodium Acid Phosphate.*—As a check on the determination of sodium acid phosphate through the estimation of total phosphoric acid by the magnesium pyrophosphate method as given above, the acidity was approximately determined by titration with normal potassium hydroxid in the presence of sodium chlorid, using phenolphthalein as indicator. The result was calculated to sodium acid phosphate. While the method is known not to be an accurate one for the titration of phosphoric acid, it was described in the U. S. P. VIII, and is considered to be sufficiently trustworthy for approximate determinations. As used in these analyses it is as follows:

To 10 c.c. of the preparation 5 gm. of sodium chlorid and a few drops of phenolphthalein solution were added. Sufficient normal potassium hydroxid solution was then run in with shaking to produce a pink color.

Ten c.c. of the material required 8.41 c.c. of normal potassium hydroxid, equivalent to 10.09 gm. of anhydrous sodium acid phosphate per 100 c.c. A duplicate required 8.61 c.c. of tenth-normal potassium hydroxid, equivalent to 10.34 gm. of sodium acid phosphate per 100 c.c. Average: 10.22 gm. of anhydrous sodium acid phosphate per 100 c.c., equivalent to 11.75 gm. of hydrated sodium acid phosphate ( $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ ) per 100 c.c. of material.

*Sodium.*—The above-described methods were approximately checked by determining the total sodium in the preparation as sodium sulphate and calculating to sodium acid phosphate. As used the method is as follows:

To a measured portion of the diluted solution an excess of barium chlorid solution was added together with sufficient ammonia water to render the mixture faintly alkaline. After standing over night, the precipitated barium phosphate was removed by filtration, the precipitate washed and the excess of barium salts in the filtrate removed by adding an excess of sulphuric acid. After

standing the barium sulphate was removed by filtration and the filtrate evaporated to dryness. The residue was heated, cooled and weighed as sodium sulphate.

From 25 c.c. of the diluted solution, equivalent to 2.5 c.c. of the original material, 0.1396 gm. of sodium sulphate was obtained, equivalent to 9.438 gm. per 100 c.c. of anhydrous sodium acid phosphate. This is equivalent to 10.85 gm. of hydrated sodium acid phosphate per 100 c.c. It should be noted that the results were somewhat lower by the sulphate method than by the methods previously given.

*Nitrogen*.—Nitrogen was determined by the Kjeldahl-Gunning method and the results calculated to hexamethylenamin. The ammonia from 25 c.c. of the diluted solution, equivalent to 2.5 c.c. of original material, required 8.69 c.c. of normal hydrochloric acid, equivalent to 12.095 gm. of hexamethylenamin per 100 c.c.

*Glycerol*.—An approximate estimation of glycerol was made by stirring some of the evaporated residue repeatedly with chloroform and filtering. The solvent was evaporated, the residue taken up in alcohol, and one fourth its volume of ether added. After standing the mixture was filtered, the filtrate was evaporated on a slowly simmering steam bath and the residue weighed. Five c.c. of the material gave a residue of 1.129 gm. of glycerol, or about 22.58 gm. per 100 c.c.

*Formaldehyd*.—The amount of formaldehyd present in Elixir Novo-Hexamine was roughly estimated as follows:

One c.c. of the material was diluted to 100 c.c. and the color from 2 c.c. of this dilution after treatment with Jorissen's reagent compared with the color produced by 2 c.c. of 1-100,000 solution of formaldehyd after treatment with the same reagent.<sup>1</sup> The method of applying the Jorissen test consisted in mixing 2 c.c. of the diluted formaldehyd solution with 1 c.c. of the Jorissen reagent, and, after thorough shaking for one-half minute, dilution of the mixture to 50 c.c. In an intitial experiment by this test the diluted Elixir Novo-Hexamine was found to be considerably too strong; therefore in succeeding

1. This reagent is prepared by dissolving 0.3 gm. of phloroglucin in 30 c.c. of 10 per cent. sodium hydroxid solution. The reagent keeps well. (Jorissen: Bull. soc. chim. Belg., 1897 [8], XI, 12 and 211).

tests the quantity of the dilution used was reduced gradually until a test using 1.2 c.c. of the dilution exactly equaled in color that from 2 c.c. of the 1-100,000 solution of formaldehyd. By calculation this result indicates that Elixir Novo-Hexamine contains about one part of free formaldehyd in 416.66 c.c.

*Synthetic Elixir Novo-Hexamine.*—A mixture was prepared in imitation of Elixir Novo-Hexamine by dissolving 12.095 gm. of hexamethylenamin, 11.16 gm. of sodium acid phosphate and 15 c.c. of U. S. P. glycerin with sufficient "sarsaparilla flavor," caramel color and water to make 100 c.c. After standing about two weeks this mixture was tested for free formaldehyd by the colorimetric method described above, and the content of free formaldehyd was found to be about one part in 416.66 c.c.

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#### "FREEZE-PROOF"

A number of requests for information were sent to THE JOURNAL of the American Medical Association concerning Johnson's "Freeze-Proof," a substance widely advertised as a reliable automobile accessory. THE JOURNAL invoked the aid of the Chemical Laboratory. An original package of "Freeze-Proof," sold by Johnson and Son, Racine, Wis., contained about  $6\frac{1}{2}$  pounds of coarse, gray, hard lumps which were almost completely soluble in water. The solution was alkaline to litmus. Qualitative tests showed the presence of a large amount of calcium and chlorid, and a very small amount or trace of aluminum, iron, magnesium, sodium, calcium and carbonate.

Quantitative determinations for the calcium and chlorid yielded the following data:

Calcium (Ca) .....	25.93
Chlorid (Cl) .....	47.1

As the calcium and chlorid are in fairly close molecular agreement, it may be stated that Johnson's "Freeze-Proof" contains 69.3 per cent.—in round numbers 70 per cent.—of anhydrous calcium chlorid. As ordinary calcium chlorid contains a large amount of moisture, it is possible that Johnson's "Freeze-Proof" is merely a form of commercial calcium chlorid.

### Details of Analysis

With the aid of a small amount of nitric acid, 3.5753 gm. of a representative sample was dissolved in water diluted to 500 c.c. Aliquot portions were used for quantitative determinations.

*Calcium.*—The calcium was determined by the ammonium oxalate method, the calcium oxalate being filtered, washed well, dried, strongly ignited and weighed (with due precautions so as not to absorb carbon dioxid and water) as calcium oxid.

(a). Twenty-five c.c. of the solution yielded 0.1250 gm. of calcium oxid, CaO. This is equivalent to 47.09 per cent. of calcium ( $\text{Ca}^{++}$ ).

(b). Twenty-five c.c. yielded 0.1255 gm. of calcium oxid. This is equivalent to 47.11 per cent. of calcium ( $\text{Ca}^{++}$ ).

*Chlorid.*—This was determined by the usual method of precipitating the silver chlorid with silver nitrate in the presence of nitric acid, and weighing as silver chlorid.

(a). Twenty-five c.c. yielded 0.6811 gm. of silver chlorid,  $\text{AgCl}$ . This is equivalent to 47.10 per cent. of chlorid ( $\text{Cl}^-$ ).

(b). Twenty-five c.c. yielded 0.6811 gm. of silver chlorid,  $\text{AgCl}$ . This is equivalent to 47.10 per cent. of chlorid ( $\text{Cl}^-$ ).

*Alkali Sulphates.*—The filtrate from the calcium determination (a) was evaporated almost to dryness, washed into a platinum dish, treated with a slight excess of sulphuric acid, and the alkali sulphates weighed. The amount found was 0.0344 gm., which showed the absence of appreciable amounts of sodium chlorid.

### DR. HAND'S WORM ELIXIR FOR CHILDREN

Dr. Hand's Worm Elixir for Children is put on the market by Smith, Kline and French Company, Philadelphia, under the name Hand Medicine Company. A specimen of this preparation was examined. The label states that 7 per cent. of alcohol is present, and that the alcohol is essential as a solvent and preservative. The statement is also made that

"This worm medicine is combined with a physic, and requires no after dose of castor oil."

Further than this, no information concerning the composition of the preparation was given on the labels or in the circulars accompanying the package.

The preparation is a thick, brownish syrup having a sweet taste and an odor resembling a mixture of anise, peppermint, sassafras and wintergreen. The specimen contained considerable insoluble matter. Under a magnifying glass this was seen to be in the form of yellowish and greenish-yellow masses.

Qualitative tests indicated the presence of santonin, gum-like substances, alcohol, reducing sugars and extractives from some emodin-bearing drug, probably senna. Other laxative substances were not found. If present their quantities must be small. Purgative salts such as Glauber's salt, Epsom salt, etc., were not present. Pinkroot and pomegranate (or their extractives) were absent. The quantitative examination was conducted with reference to the santonin content only. The preparation was found to contain about 0.2 gm. of santonin per 100 c.c., equivalent to about 0.91 grain per fluidounce. Each drachm (teaspoonful) of the preparation contains a little more than  $\frac{1}{10}$  of a grain of santonin. Several years ago, Dr. Hand's Worm Elixir was examined in the laboratory of the Connecticut Agricultural Experiment Station (Rep. Conn. Agric. Exp. Sta., 1914, p. 325). At that time the presence of 0.482 gm. of santonin (2.2 grains per fluidounce) was reported.

#### Details of Analysis

*Santonin.*—Owing to the presence in the preparation of considerable quantities of gummy substances which prevented the collection of the undissolved santonin by filtration, the method used in this laboratory some time ago for the detection of santonin in Thacher's Worm Syrup (Rep. Chem. Lab., A. M. A., 1911, 4, 90) could not be employed. Accordingly, the gum-like substances were precipitated by pouring a weighed quantity of the material, with agitation, into a large excess of warm alcohol. After standing over night, the mixture was filtered, and, after the addition of water, the filtrate was evaporated to remove the alcohol. The precipitate of gum-like substances was dissolved in water and a large excess of alcohol added. After standing the mixture was filtered, the filtrate evaporated to small volume and the residue added to the residue from the filtrate obtained from the first precipitation and filtration. The united residues formed a turbid solution. This was shaken with chloroform, the solvent evaporated, and the residue treated by the Katz

method, slightly modified (*Arch. Pharm.*, **237**, 245, 1899), for the determination of santonin. Briefly, the method as carried out is as follows:

The chloroform extract was heated with 100 c.c. of saturated barium hydroxid solution, the mixture filtered while hot, the resin-like residue washed repeatedly with hot barium hydroxid solution and the washings decanted through the filter. The filter paper was then placed in the beaker in which the residue had been treated with the barium hydroxid solution, 30 c.c. of alcohol added, the mass acidified with hydrochloric acid, the mixture warmed and filtered. The filtrate was evaporated nearly to dryness, the residue treated with barium hydroxid solution as above described, and the filtrate added to the filtrate earlier obtained. The excess of barium hydroxid was removed from the united filtrates by treatment with a stream of carbon dioxid and removal by filtration of the barium carbonate formed. The precipitated barium carbonate was washed on the filter and the washings and the filtrate united. The solution was evaporated to about 20 c.c., slightly acidified with dilute hydrochloric acid and the liberated santoninic acid extracted with chloroform. The solvent was evaporated, the residue boiled with 50 c.c. of 15 per cent. alcohol, the mixture filtered and the filtrate allowed to cool. After standing over night, the crystals of santoninic acid were collected in a weighed Gooch erueible, dried at 60 degrees and weighed. The weight found is multiplied by 0.931 to obtain the weight of santonin originally present in the preparation.

*Senna*.—Some of the preparation was poured, with constant stirring, into a large excess of warm alcohol, which had been slightly acidified with hydrochloric acid. The mixture was allowed to stand in a closed vessel for forty-eight hours during which most of the gummy substances separated. The supernatant alcoholic liquid was then poured through a filter, one half its volume of water added, the solvent evaporated until the alcohol was nearly removed, the residue shaken with ether and the ether solution washed with water. The ether solution did not fluoresce, indicating the probable absence of extractives from cascara and aloes. A portion of the ether layer was shaken with very dilute ammonia water. A pink color in the aqueous layer pointed toward the presence of an

emodin-bearing drug. Another portion of the ether solution was shaken with a saturated solution of borax. A rose color was produced. This color is said by Hubbard (*Jour. Indust. and Engin. Chem.*, 1917, ix, 518) to be given by rhubarb under these conditions, but not by senna, the latter giving either no color or a light brown color. Nevertheless, after subjection to the treatment above outlined, a known solution of senna gave a pink color. The absence of rhubarb, however, was shown by shaking a portion of the ether extract with a saturated solution of ferrous sulphate. No blue color resulted.

*Purgative Salts.*—Some of the material was evaporated to dryness and the residue burned. Only small amounts of ash were produced. The absence, at least in therapeutically effective amounts, of the heavy purgative salts, such as Rochelle salt, Epsom salt, etc., was thus demonstrated.

*Alkaloids.*—The chloroform extract from the preparation was dissolved in warm barium hydroxid solution and the mixture shaken with chloroform. The solvent was drawn off, evaporated, the residue taken up in dilute hydrochloric acid and the solution treated with iodin solution. Only a mere trace of precipitate was given. After this had stood over night the mixture was filtered, the precipitate decomposed with sodium sulphite solution, the resultant solution made alkaline and shaken with chloroform. The solvent was evaporated, the residue taken up in dilute hydrochloric acid, and the solution tested for alkaloids with iodin solution. No precipitate was given. From this it was concluded that such alkaloids as caffeine and colchicine, which are extracted by chloroform from slightly acid solution, if present at all in Dr. Hand's Worm Elixir, must be contained in that preparation in very small amounts. The material from which the santonin had been extracted was made alkaline with ammonia water and the solution extracted with chloroform. The solvent was evaporated in presence of a little dilute hydrochloric acid, the residue taken up in water and an excess of iodin solution added. A faint precipitate was produced. After this had stood over night the mixture was filtered, the precipitate decomposed with sodium sulphite solution, the resultant solution made alkaline and shaken with chloroform. The solvent was evaporated, the residue taken up in dilute hydrochloric acid and the solution tested for alkaloids with iodin solution. No precipitate was given. These tests were

taken to indicate the absence of alkaloids in Dr. Hand's Worm Elixir. Pinkroot (*Spigelia marilandica*) and pomegranate (*Punica granatum*), two anthelmintic drugs which possibly might have been expected to be present in a preparation of this class, contain alkaloids. Consequently these two drugs or their extractives are not present in Dr. Hand's Worm Elixir.

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### IODEOL AND IODAGOL

Iodeol and Iodagol (formerly called Iodargol) are said to be preparations of "colloidal iodin." They are the products of Viel and Company, Rennes, France. For the past three years they have been sold and extensively advertised in the United States by David B. Levy, Incorporated, New York.

Iodeol and Iodagol are put up in a number of forms, for instance:

"Iodeol Ampoules each containing 1 c.c. (20 centigrammes colloidal iodine in an oily vehicle.)"

"Iodeol External, containing 50 per cent. colloidal iodine."

"Iodagol Ampoules, each containing 2 c.c. (50 centigrammes colloidal iodine in an oily vehicle.)"

The advertising propaganda for Iodeol and Iodagol is based on the claim that, the iodin being in the colloidal state, it has the properties of free iodin while the preparations may yet be used in concentrations and under conditions which would make the use of ordinary free iodin impossible.

Iodeol and Iodagol were submitted to the Council on Pharmacy and Chemistry with the statement that each was a "suspension of electro-chemical colloidal iodine in a vehicle of purified oil." It was stated that the difference between Iodeol and Iodagol "consists in the minuteness of the grains and the content of iodine," the particles of iodin in Iodagol being "coarser than those of Iodeol." It was also stated that "Viel's method of producing colloidal iodine consists in dissociating metalloidal iodine by an electric current, the product being then rendered stable by suspension in a neutral oil, which is washed in alcohol, sterilized, and saturated with a hydro-carbon (a camphene) to prevent the iodine from combining with the fatty body."

As the information in regard to the composition and character of Iodeol and Iodagol was unsatisfactory, and requests for more definite information brought no satisfactory replies, this laboratory was requested to examine the preparations,

particularly in reference to the claimed iodin content, and also, if possible, in regard to the condition of the contained iodin.

#### IODIN CONTENT OF IODEOL AND IODAGOL

To determine the iodin content of the specimens of Iodeol and Iodagol submitted to the Council, the following methods were used: (1) the Carius method; (2) the lime method;<sup>1</sup> (3) Kendall's method, as described for larger amounts of iodin;<sup>2</sup> (4) Pringsheim's method, involving fusion with sodium peroxid,<sup>3</sup> and (5) the saponification method, as described in the Reports of the A. M. A. Chemical Laboratory (1916, **9**, 118), involving the saponification of the material with alcoholic potash, evaporation of the solution, and charring of the residue, after which the charred mass is leached out with water and the iodin precipitated by silver nitrate.

A. Iodeol, for Intramuscular Injection. The sample contained in 1 c.c. ampules and was stated to contain 20 per cent. of iodin in colloidal condition.

1. CARIUS METHOD.—(a) Iodeol, 0.2589 gm., yielded 0.0302 gm. silver iodid equivalent to 0.0212 gm. iodin, or 8.19 per cent.

(b) Iodeol, 0.2085 gm., yielded 0.0318 gm. silver iodid equivalent to 0.0172 gm. iodin, or 8.25 per cent.

3. KENDALL'S METHOD.—(a) Iodeol, 1.052 gm., required 39.25 c.c. N/10 thiosulphate in the titration of the iodin liberated by the iodate formed from the iodin present in the sample. This is equivalent to an iodin content of 0.0830 gm., or 7.89 per cent.

(b) Iodeol, 0.9041 gm., required 34.25 c.c. N/10 thiosulphate solution for the titration of the iodin liberated by the iodate formed. This is equivalent to 0.0724 gm. iodin, or 8.01 per cent.

(c) Iodeol, 1.028 gm., required 37.55 c.c. N/10 thiosulphate solution, equivalent to 0.0794 gm. iodin, or 7.73 per cent.

1. Treadwell: Quantitative Analysis, translated by Hall, Ed. 4, 1915, p. 329.

2. Jour. Am. Chem. Soc., 1912, **34**, 894.

3. Ber. d. deutsch. chem. Gesellsch., **36**, 4244.

B. Iodeol for External Use Only. The sample was contained in a small glass stoppered bottle carefully sealed. It was stated to contain 50 per cent. of iodin.

1. CARIUS METHOD.—(a) Iodeol, 0.2609 gm., yielded 0.1097 gm. silver iodid, equivalent to 0.05935 gm. iodin, or 22.75 per cent. The silver iodid after solution in potassium cyanid was electrolyzed and the deposited silver weighed, 0.0506 gm. metallic silver being obtained, which corresponded to 0.1101 gm. silver iodid.

(b) Iodeol, 0.2529 gm., yielded 0.1072 gm. silver iodid, equivalent to 0.05795 gm. iodin, or 22.91 per cent. This precipitate on electrolysis yielded 0.0483 gm. silver, equivalent to 0.1052 gm. silver iodid.

2. THE LIME METHOD.—(a) Iodeol, 0.4823 gm., yielded 0.1850 gm. silver iodid, equivalent to 0.1000 gm. iodin, or 20.73 per cent.

(b) Iodeol, 0.4731 gm., yielded 0.1807 gm. silver iodid, equivalent to 0.0977 gm. iodin, or 20.65 per cent.

3. KENDALL'S METHOD. (a) Iodeol, 1.036 gm., required 41.25 c.c. N/10 thiosulphate solution in the titration of the iodin liberated by the iodate formed. This is equivalent to 0.0873 gm. iodin, or 8.43 per cent.

(b) Iodeol, 1.029 gm., required 43.15 c.c. N/10 thiosulphate in the titration, equivalent to 0.0913 gm. iodin, or 8.87 per cent.

4. PRINGSHEIM'S METHOD.—(a) Iodeol, 0.4873 gm., yielded 0.1011 gm. silver iodid, equivalent to 0.0546 gm. iodin, or 11.40 per cent.

(b) Iodeol, 0.5232 gm., yielded 0.1102 gm. silver iodid, equivalent to 0.0596 gm. iodin, or 11.39 per cent.

5. SAPONIFICATION METHOD. A sample of Iodeol weighing 1.1402 gm. yielded 0.1950 gm. silver iodid, equivalent to 0.1052 gm. iodin, or 9.23 per cent.

C. Iodagol. This sample was contained in a glass stoppered bottle which had been carefully sealed. It was stated to contain 25 per cent. of iodin.

1. CARIUS METHOD.—(a) Iodagol, 0.2595 gm., yielded 0.0530 gm. silver iodid, equivalent to 0.0287 gm. iodin, or 11.05 per cent. The silver iodid when dissolved in potassium cyanid and electrolyzed yielded 0.0243 gm. metallic silver, equivalent to 0.0530 gm. silver iodid.

(b) Iodagol, 0.2922 gm., yielded 0.0596 gm. silver iodid, equivalent to 0.0322 gm. iodin, or 11.03 per cent.

The metallic liver obtained by electrolyzing the solution of the silver iodid in potassium cyanid weighed 0.0273 gm., equivalent to 0.0594 gm. silver iodid.

2. THE LIME METHOD.—(a) Iodagol, 0.610 gm., yielded 0.1163 gm. silver iodid, equivalent to 0.0629 gm. iodin, or 10.15 per cent.

(b) Iodagol, 0.4809 gm., yielded 0.0923 gm. silver iodid, equivalent to 0.0499 gm. iodin, or 10.37 per cent.

*Discussion.*—The results clearly show that in Iodeol for External Use, Kendall's method, Pringsheim's method

TABLE 1.—SUMMARY OF THE DETERMINATION OF IODIN IN IODEOL AND IODAGOL

IODEOL (EXTERNAL) (50%)

Method	Carius	Lime	Kendall	Pringsheim	Saponification
1st test .....	8.19	.....	7.89	.....	.....
2d test .....	8.25	.....	8.01	.....	.....
3d test .....	.....	.....	7.73	.....	.....
Average .....	8.22	.....	7.88	.....	.....

IODEOL (EXTERNAL) (50%)

Method	Carius	Lime	Kendall	Pringsheim	Saponification
1st test .....	22.75	20.73	8.43	11.40	9.23
2d test .....	22.91	20.65	8.87	11.39	.....
Average .....	22.83	20.69	8.65	11.40	.....

IODAGOL (25%)

Method	Carius	Lime	Kendall	Pringsheim	Saponification
1st test .....	11.65	10.15	.....	.....	.....
2d test .....	11.03	10.37	.....	.....	.....
Average .....	11.04	10.26	.....	.....	.....

and the saponification method all give untrustworthy results. Kendall's method particularly gave widely differing results on these preparations, the reason for which could not be discovered in any manipulative error. The results obtained by the Carius method may be depended on as essentially correct. The analyses check closely and the appearance of the assay was normal, and on examination of the tube with a hand glass no crystals of iodin could be discovered. The results by the lime method check roughly with those obtained by the Carius method. That these results are somewhat lower may be accounted

for by the fact that the neutralization of the lime by dilute nitric acid might have given occasion for a slight oxidation and loss of iodin. The electrolytic determinations of silver in the silver iodid precipitate show that iodin is the sole halogen present.

#### NATURE OF THE IODIN COMPOUND IN IODEOL AND IODAGOL

On steam distillation of the Iodeol for External Use, about 70-75 per cent. came over with the steam.

Preliminary tests indicated the presence in this distillate of guaiacol, and the odor suggested the presence of camphor also, but it was not identified as a constituent. The greater part—at least 90 per cent. of the volatile matter—is an oil heavier than water, which, on heating for the purpose of getting the boiling point, decomposed with some violence at 188 C., liberating iodin. It seems, then, that this body contains the iodin.

It was found that when iodin is added to eucalyptus oil, the cineol iodid described in the literature is formed (greenish and crystalline).

However, when the eucalyptus oil was added to an excess of iodin, a very vigorous reaction ensued, giving a brownish oil very much like the sample of Iodeol. On steam distillation this gave an oil heavier than water which on heating decomposed at 190-192 C., liberating iodin.

This renders it quite probable that the majority of the volatile constituents of Iodeol for External Use is such an iodin compound of cineol.

The material of the Iodeol sample not volatile with steam consisted of a brown petrolatum-like material, which does not seem to be at all saponifiable.

In regard to the behavior of the distillate from Iodeol (for external use only) with acid nitrite, it was found that in the case of the freshly distilled product, when treated with the acid nitrite mixture and starch added, no blue color results immediately, but that a distinct color can be noted in one-half to one hour which grows deeper on standing.

The residue left after steam distillation seems to be free from iodin. On treating this residue with an excess of alcoholic potash (it is almost completely unsaponifiable), and on charring, leaching and acidifying the filtered solution, no test for iodin could be obtained. It is possible that iodin is lost in the charring process, but extremely unlikely that all should be lost.

## REPORT OF COUNCIL REFEREE

The findings of the Laboratory were sent to the American agent for transmission to E. Viel and Company. After a lengthy delay a reply was received.

The following confirmation of the Laboratory's findings by the referee of the Council on Pharmacy and Chemistry in charge of Iodeol and Iodagol and his discussion of the above mentioned reply is taken from the report of the Council declaring Iodeol and Iodagol falsely labeled. This report also includes a report of the pharmacologic, bacteriologic and clinical investigation made by or under the direction of the referee.

The examination at the Chemical Laboratory of the American Medical Association, as well as that of the referee, shows that the various samples of Iodeol and Iodagol examined contained a little less than one half of the total iodin claimed. These facts were reported to the American agent. After a lengthy delay a reply was received which presented a double excuse: (1) that the full amount of iodin had been added, whatever had become of it later; (2) that the claims were made for "colloidal iodin" and that this is not elementary iodin in the colloidal state, but a preparation of iodin containing only 50 per cent. of real iodin. Neither explanation can be taken seriously, as they are obvious quibblings. The referee concludes that the preparations are falsely labeled as to iodin content.

In the information sent the Council, Iodeol and Iodagol were defined as "a suspension of electro-chemical colloidal iodin in a vehicle of purified oil." Numerous inquiries have failed to elicit more specific information from the manufacturer or his agent. The statement of composition can mean only that the preparations contain free iodin (but in colloidal form) suspended in oil. No evidence to substantiate this claim has been submitted. (There is evidence that the preparations contain colloidal particles, but it does not indicate if this colloidal material is iodin, or a combination of iodin or indeed whether the colloid component contains any iodin.) The recent statements of the agent seem to concede that what they call "electro-colloidal iodin" contains only about 50 per cent. of real iodin, in other words, that it is not "colloidal iodin" at all but a mixture or combination of iodin with some other unnamed substance. This, of course, is something very different.

Certain results reported from the American Medical Association's Chemical Laboratory suggest that the so-called "colloidal iodin" of Iodeol may be a combination of iodin with a volatile oil. The investigations of the referee indicate that the iodin exists in a rather resistant form or combination behaving altogether differently from ordinary free iodin, and rather resembling the behavior of iodin substitution products, such as iodized fats or phenols. Briefly, then, the recent admissions of the agents indicate that Iodeol does not contain "colloidal iodin" in a chemical sense, and there are indications that it does contain its iodin in a rather firm (chemical) combination.

From a study of different specimens of Iodeol, the referee concludes that fresh specimens contain no free iodin, and that old ones contain small amounts as a result of decomposition. Iodeol has the solubility characteristics of fats and fat-like compounds. The examination, as a whole, shows that Iodeol contains a peculiar and rather resistant form or combination of iodin. There is nothing in the chemical data that suggests that it could act differently from ordinary iodin compounds, such as iodized fats. It would not act as ordinary iodin.

#### AGENT DISCONTINUES SALE OF PRODUCTS

The report of the Council was brought to the attention of the American agent, David B. Levy, Incorporated, and through this firm to the French manufacturers, E. Viel and Company. The manufacturers having refused to modify their claims regarding the iodin content of their products, the firm of David B. Levy, Incorporated, advised the Council that it had decided to sever its connection with these products and to discontinue their sale. The letter announcing this decision is quoted below, for the reason that it emphasizes the difficulties encountered and the risks assumed when American firms accept the agency for foreign pharmaceutical products, particularly when such agents have no technically trained men in their employ.

(9/6/17) "We acknowledge with thanks receipt of yours of recent date with copy of decision of the Council. Immediately after receipt of same the Board of Directors of our corporation have taken up and finally gone most seriously into the question of Iodeol and Iodagol.

"For three years we have been spending large sums of money in advancing the sale and field for the said

products and have based and placed our time, money and labor upon the scientific claims of the manufacturer.

"In view of the fact that the Department of Agriculture has consistently sustained the position urged by the American Medical Association, with respect to the Iodine content, and that the manufacturer, although repeatedly urged and entreated by us to sustain his products with respect to content, as set forth in his literature, has either been unwilling or unable to do so, our concern has therefore come to the conclusion that it is better to lose our money than to have our business integrity and reputation doubted, and henceforth will discontinue the sale of the products in question.

"This matter has been pending over a long period of time, and while the decision reached is one entailing enormous loss, we wish to state that we appreciate your stand, and the many courtesies that the Council and yourself have extended us in the past."

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#### IODOLENE AND THE SOLUBILITY OF IODIN IN LIQUID PETROLATUM

The Council on Pharmacy and Chemistry was asked to examine a preparation submitted with the statement that it was "iodin crystals incorporated in a petroleum product." The name "Iodolene" was proposed by the promoters, providing the product was found eligible for New and Nonofficial Remedies.

Iodolene was stated to have been prepared by treating a Liquid Petrolatum, obtained from Gulf Coast petroleum, with an excess of iodin; the mixture was subsequently "placed in an oven for three hours." The claim was made that this method of procedure produced a preparation containing more iodin than market specimens which had been examined, namely: "over 1.50 per cent. free iodine."

Two specimens of the product were submitted, one stated to have been unfiltered, and the other filtered. Both of the specimens emitted a strong odor of hydrogen sulphide upon removing the stopper from the respective containers.

*Iodin Content of Iodolene.*—The iodin content of the filtered specimen was determined thus: A weighed amount—3 to 5 gm.—was transferred to a separator by means of 20 c.c. of ligroin, used in portions. Twenty c.c. of 10 per cent. potassium iodid solution was added and the free iodin titrated

with tenth-normal sodium thiosulphate solution (with agitation), the end point being the absence of a yellow color in the *aqueous* layer. The amount of free iodin was found to be 1.32 per cent.

*The Solubility of Iodin in Liquid Petrolatum.*—To determine the solubility of iodin in Liquid Petrolatum, 200 c.c. of Liquid Petrolatum-Squibb (said to be composed of hydrocarbons of the naphthene series) and 200 c.c. of Stanolind Liquid Paraffin (said to be composed chiefly of marsh gas hydrocarbons) were each treated with 5 gm. of iodin crystals. The two mixtures were maintained for a week at a temperature somewhat above that of the room and agitated occasionally. Each was then cooled to room temperature (about 22 C.), agitated for a day and then filtered. The amount of iodin in the preparation made with Liquid Petrolatum-Squibb was found to be 1.42 per cent. The iodin content of the preparation made with Stanolind Liquid Parafin was 1.30 per cent.

In view of these findings the prospective manufacturer was advised that the Council cannot countenance a proprietary name for an unofficial, simple solution of iodin in liquid petrolatum.

#### KAR-RU

A package of "Kar-Ru" "Prepared by the Kar-Ru Chemical Co., Tacoma, Wash.," retail price \$5.00, was sent to the Chemical Laboratory for an opinion regarding its composition. The label on the Kar-Ru box declared that

"It is effective in Kidney, Liver, Bladder, Stomach, and Catarrhal Troubles, Mental and Physical Debility, Neuritis, Eczema, Blood Diseases, Irregular Menstruation, and the most Acute and Chronic Rheumatic Afflictions."

The printed matter accompanying Kar-Ru gives no suggestion or clue in regard to the composition of the forty powders, numbered consecutively from 1 to 40, contained in the Kar-Ru box.

A number of the powders were selected at random for the qualitative tests. The material had a gray color, and was odorless. Tests indicated the presence of corn starch, sugar, and charcoal. Substances, the presence of which might be expected in such a remedy, such as alkaloids, heavy metals, salicylates, iodids and hexamethylenamine were tested for and not found.

### MAGNESIUM ACID CITRATE—"CITRESIA"

A specimen of "Citresia," said by the manufacturer, Horace North, New York, to be a hydrated magnesium acid citrate was submitted to the Council on Pharmacy and Chemistry. The product was said to crystallize with five molecules of water of hydration, the formula claimed for the substance being  $MgHC_6H_5O_7 \cdot 5H_2O$ . The manufacturer stated that magnesium acid citrate is a newly discovered substance. A number of acid citrates of magnesium are described in the literature, but none with the formula claimed for Citresia. The specimen examined was a fine, faintly yellowish-white, crystalline, odorless powder having an acid taste. A specimen of the material in the form of large, pale yellowish crystals was also sent by the manufacturer. This was not submitted to analysis. The material was found to be very soluble in water, but practically insoluble in alcohol. The analysis indicated that the preparation has essentially the composition claimed for it.

Citresia was accepted by the Council on Pharmacy and Chemistry for inclusion in New and Non-official Remedies.

#### Details of Analysis

*Water.*—Water was determined by prolonged drying at 175 C. A loss of 29.7 per cent. was found whereas theory for the formula given by the manufacturer requires 29.58 per cent. From 1.0008 gm. of material a loss of 0.2971 gm. was found, equivalent to 29.68 per cent. From 1.0019 gm. of material a loss of 0.2978 gm. was found, equivalent to 29.72 per cent. Average, 29.70 per cent. of loss on drying.

*Ash.*—This was determined in the usual way. From 1.0008 gm. of material 0.1391 gm. of ash was obtained, equivalent to 13.72 per cent.

*Alcohol Soluble Material.*—A weighed quantity of the material was shaken with about five times its weight of alcohol, the mixture filtered, the filtrate evaporated to dryness, the residue dried at 100 C. and weighed. From 5.0029 gm. of material a residue of 0.0131 gm. was obtained, equivalent to 0.26 per cent. This residue had an acid reaction, did not contain magnesium, and responded to tests for citric acid.

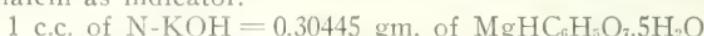
*Magnesium.*—This was determined by precipitation with sodium phosphate solution in the presence of ammonium

chlorid and ammonia, solution of the precipitate in dilute hydrochloric acid, reprecipitation in presence of ammonia, and weighing as magnesium pyrophosphate in the usual way.



From 0.7033 gm. of material 0.2571 gm. of magnesium pyrophosphate was obtained, equivalent to 0.056243 gm. of magnesium, or 99.94 per cent. of hydrated magnesium acid citrate,  $\text{MgHC}_6\text{H}_5\text{O}_7 \cdot 5\text{H}_2\text{O}$ . From 0.6953 gm. of material 0.2545 gm. of magnesium pyrophosphate was obtained, equivalent to 0.055674 gm. of magnesium, or 100.07 per cent. of hydrated magnesium acid citrate. Average, 100.01 per cent. of hydrated magnesium acid citrate.

**Hydrated Magnesium Acid Citrate.**—This was estimated by titration with normal potassium hydroxid, using phenolphthalein as indicator.



A weight of 2.9760 gm. of material required 9.838 c.c. of normal sodium hydroxid for neutralization, equivalent to 2.995179 gm. of hydrated acid magnesium citrate, or 100.67 per cent. A weight of 2.9882 gm. of material consumed 9.888 c.c. of normal sodium hydroxid, equivalent to 3.0104 gm. of hydrated acid magnesium citrate or 100.74 per cent. Average, 100.7 per cent. of hydrated acid magnesium citrate.

### MINERAL SALTS

One box of "Mineral Salts" (priced \$1), sold by the Franklin Medicine Company, 3252 Wallace Street, Chicago, was submitted to the Chemical Laboratory for examination. After directing how to prepare the solution of the "salts," the label on the box gave the following:

For Catarrh: Snuff, gargle and drink water three times a day.

For Sore Throat: Gargle and place Salts on tongue, dry also snuff Salts, dry for Catarrh.

The box contained about 36 gm. ( $1\frac{1}{2}$  ounces) of a white, odorless powder, having a taste suggesting both sodium chlorid and sodium bicarbonate. Qualitative tests demonstrated the presence of sodium, potassium (trace), bicarbonate, chlorid and a slight trace of organic matter. Tests for sulphate, phosphate, and heavy metals were negative. Quantitative determinations yielded the following:

Sodium ( $\text{Na}^+$ ) .....	36.19 per cent.
Bicarbonate ( $\text{HCO}_3^-$ ).....	15.31 per cent.
Chlorid ( $\text{Cl}^-$ ) .....	46.62 per cent.

From these figures the following composition may be adduced:

Sodium bicarbonate .....	21.07 per cent.
Sodium chlorid .....	79.94 per cent.

In other words, a mixture essentially similar in composition and amount to "Mineral Salts" may be compounded by mixing one-half tablespoonful (two teaspoonfuls) of baking soda with two tablespoonfuls of ordinary salt. The cost of such a mixture would not exceed 1 cent.

#### Details of Analysis

*Sodium*.—About 0.6 gm. of the sample, accurately weighed, was placed in a previously weighed platinum dish, and about 4 c.c. of concentrated sulphuric acid added. The mixture was heated on the steam bath for one-half hour, to remove the gaseous  $\text{CO}_2$  and HCl. It was then transferred to a triangle, and carefully heated to expel all the sulphuric acid. It was then heated over a Meeker burning for fifteen minutes, cooled, and then weighed as  $\text{Na}_2\text{SO}_4$ . (a) 0.6180 gm. of the sample yielded 0.0008 gm. of  $\text{Na}_2\text{SO}_4$ . This is equivalent to 36.23 per cent. Na. (b) 0.5174 gm. of the sample yielded 0.5703 gm. of  $\text{Na}_2\text{SO}_4$ . This is equivalent to 35.77 per cent. Na. (c) 0.8123 gm. of the sample yielded 0.9053 gm. of  $\text{Na}_2\text{SO}_4$ . This is equivalent to 36.15 per cent. Na.

*Bicarbonate*.—The bicarbonate was determined volumetrically. The sample, accurately weighed, was placed in a 250 c.c. Erlenmeyer flask, 100 c.c. of water added, and titrated with normal hydrochloric acid, using methyl orange as an indicator. As soon as the first end-point was reached, the solution was boiled for two minutes, and titrated again to end-point. (a) 2.1206 gm. required 5.32 c.c. N/1 HCl. This is equivalent to 21.07 per cent. of  $\text{NaHCO}_3$  or to 15.31 per cent. of  $\text{HCO}_3^-$  or  $\text{HCO}_3$ . By difference, the amount of sodium in the sodium bicarbonate present would be 5.76 per cent. Na. (b) 2.3157 gm. of the sample required 5.81 c.c. N/1 HCl. This is equivalent to 21.07 per cent.  $\text{NaHCO}_3$ .

*Chlorid*.—About 0.5 gm. of the sample, accurately weighed, was placed in an Erlenmeyer flask, dissolved in 100 c.c. of water and 10 c.c. of nitric acid added; after the carbon dioxide had escaped, a slight excess of silver nitrate solution was added. The flask was stoppered and vigorously shaken.

The supernatant was decanted through a Gooch crucible (previously dried and weighed). The residual silver chlorid was shaken five times with about 150 c.c. of water, acidified with nitric acid. Finally the silver chlorid was all washed on to the Gooch, dried and weighed. (a) 0.4504 gm. of the sample yielded 0.8524 gm. of AgCl. This is equivalent to 46.78 per cent. Cl. or Cl<sub>2</sub>. (b) 0.4451 gm. of the sample yielded 0.8365 gm. of AgCl. This is equivalent to 46.48 per cent. Cl.

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### STUART'S CALCIUM WAFER COMPOUND

In 1915, THE JOURNAL referred to the Laboratory an inquiry regarding the composition, particularly as to the presence or absence of arsenic, of Stuart's Calcium Wafer Compound, sold by the F. A. Stuart Company, Marshall, Mich. An examination made to determine the general character of this preparation indicated that it contained calcium sulphid and aloes or aloin as its essential ingredients. The presence of arsenic could not be demonstrated (Reports A. M. A. Chem. Lab., 1915, **8**, 126).

Recently it was brought to the attention of the Laboratory that Stuart's Calcium Wafer Compound was analyzed by the state board of health of New Hampshire in connection with the death of a child who had swallowed some of the medicine, and that strychnin had been found in it as well as in the organs of the child.

As no search for strychnin had been made in the Laboratory's examination, it was thought desirable to examine the remainder of the specimen still in the files with a view of confirming the analysis of the New Hampshire chemists and also to examine the preparation as now offered for sale.

A specimen of Stuart's Calcium Wafer Compound purchased in October, 1917, was found to consist of brown coated tablets, having an average weight of 0.0809 gm. (12<sub>5</sub> grains). A qualitative examination of the tablets (including the coating which could not be readily separated from the tablet proper) indicated the presence of calcium, sulphid, sulphate, carbonate and aloin. Strychnin or other potent alkaloids could not be detected.

It thus appears that, agreeing with the previous examination, Stuart's Calcium Wafer Compound contains calcium, sulphid and aloes or aloin as its essential constituents.

Quantitative determinations indicated that the tablets contained 11.3 per cent. of calcium sulphid (CaS), equivalent

to 16.35 per cent. of crude calcium sulphid, U. S. P., or 0.0146 gm. ( $\frac{1}{4}$  grain) per tablet.

Examination of a specimen of Stuart's Calcium Wafer Compound purchased in 1908 indicated that this contained 0.57 per cent. of strychnin, or 0.00060 ( $\frac{1}{107}$  grain) per tablet.

Examination of the specimen purchased in 1915 and previously reported on proved that this also contained strychnin. The amount found was equivalent to 0.58 per cent. of strychnin, or 0.00046 gm. ( $\frac{1}{140}$  grain) per tablet.

From the foregoing, it seems that in the past Stuart's Calcium Wafer Compound contained strychnin in amounts sufficient to make indiscriminate and unrestricted use by the public of this "patent medicine" dangerous. It further indicates that strychnin has been omitted from the tablets now sold, probably as a result of the fatality reported by the New Hampshire State Board of Health.

#### Details of Analysis

*Strychnin.*—About 2 gm. (accurately weighed) of the finely powdered tablets and 25 c.c. of water were placed in a separatory funnel, made alkaline with ammonium hydroxid, and extracted with four 20 c.c. portions of chloroform. The combined extracts were evaporated to dryness and extracted with several portions of dilute hydrochloric acid to remove resins. The acid extract was then made alkaline and again extracted with four 20 c.c. portions of chloroform, and the latter evaporated to dryness. The tests for strychnin were made on this extract. The titration of strychnin was conducted according to the method outlined under the assay for the total alkaloids in *nux vomica*, U. S. P. IX, p. 281, fiftieth-normal sulphuric acid being employed. Each cubic centimeter that was consumed corresponded to 6.68 mg. of strychnin.

1917 Sample: No strychnin could be detected in a sample purchased on the open market. In order to verify the accuracy of the method of extraction,  $\frac{1}{4}$  grain of pure strychnin sulphate was added to a sample, and the process repeated, resulting in the recovery of the total amount added.

1908 Sample: Strychnin was detected in this sample and the 1915 sample by the sulphuric acid-dichromate test. According to Autenrieth-Warren (Detection of Poisons, 1915, p. 96), "more than traces of brucine prevent the detection of strychnin with concentrated sulphuric acid and potassium

dichromate." After this statement had been confirmed by means of qualitative tests, it was concluded that the residues consisted essentially of strychnin, and consequently they were calculated as such. Twenty-five tablets weighing 2.6695 gm. were extracted according to the method outlined above, and estimated according to the U. S. P. IX method. For the back titration of the 25 c.c. of fiftieth-normal sulphuric acid added (factor 1.2112), it required 27.9 c.c. of fiftieth-normal sodium hydroxid (factor 1.006), corresponding to 2.25 c.c. of fiftieth-normal sulphuric acid consumed by the alkaloid, equivalent to 0.0150 gm. of strychnin, or 0.057 per cent.

1915 Sample: Twenty tablets weighing 1.5998 gm. required 28.7 c.c. of fiftieth-normal sodium hydroxid (factor 1.006) for back titration, corresponding to 1.4 c.c. of fiftieth-normal sulphuric acid consumed by the alkaloid, equivalent to 0.0093 gm., or 0.58 per cent.

*Sulphid.* This was determined in the 1917 sample by the iodometric cadmium sulphid method employed in the analysis of steels. A weighed quantity was treated with dilute hydrochloric acid in a generating flask in an atmosphere of hydrogen, and the evolved gas passed through wash bottles containing a solution of cadmium chlorid. The cadmium sulphid formed was filtered off, washed, suspended in 1 per cent. sulphuric acid solution, and decomposed by the addition of an excess of tenth-normal iodin solution. After standing twenty minutes the excess iodin was titrated with tenth-normal sodium thiosulphate solution. Each c.c. of tenth-normal iodin consumed equals 0.00170 gm. of hydrogen sulphid.

In this determination, 0.5015 gm. of the tablets required 15.6 c.c. of tenth-normal iodin (factor 1.089), equivalent to 0.0265 gm. of hydrogen sulphid, corresponding to 0.0563 gm. of pure calcium sulphid, or 11.23 per cent; calculated to crude calcium sulphid, U. S. P., this would be equivalent to 16.3 per cent.

In another determination, 0.4932 gm. of the wafers required 15.55 c.c. of tenth normal iodin solution (factor 1.089), equivalent to 0.0264 gm. of hydrogen sulphid, corresponding to 0.0560 gm. of pure calcium sulphid, or 11.36 per cent.; calculated as crude calcium sulphid, U. S. P., this would be equivalent to 16.4 per cent. The average of the foregoing

results is 11.3 per cent. pure calcium sulphid, or 16.35 per cent. crude calcium sulphid, U. S. P.

*Aloin*.—The aqueous extract of the tablets when filtered and heated with borax solution developed a green fluorescence, indicating the presence of aloes or aloin. This test, combined with the characteristic taste, was sufficient evidence to identify one of the constituents as aloin or aloes.



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